DETERMINATION OF THE MINERALOGICAL AND ELEMENTAL COMPOSITION OF AIRBORNE DUST FROM UNDERGROUND COAL MINES

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RÉSUMÉ

1. Le Laboratoire de recherche sur le charbon a réalisé une étude exhaustive des méthodes utilisées pour analyser la minéralogie et la composition chimique des poussières inhalables produites dans les mines de charbon. La méthode analytique la plus appropriée à la caractérisation minéralogique, morphologique et chimique des poussières de charbon inhalables consiste en la microscopie électronique à balayage combinée à la microanalyse des rayons X par spectromètre dispersif en énergie (SEM-ESX) des particules de poussière telles que captées sur les filtres.

2. Les poussières inhalables produites dans les mines de charbon de la Société de développement du Cap-Breton et recueillies sur des filtres Nuclepore ou Millipore contenaient en moyenne les proportions de minéraux suivantes, calculées en fonction de 500 points d'analyse sur 6 filtres:

	% du poids
charbon	41.94
illite	31.94
calcite	3.88
quartz	9.49
kaolinite	2.29
pyrite	2.54
sidérite	2.14
autres	5.78

3. L'analyse par SEM-EDX de 3 075 macroparticules a permis de découvrir la présence d'autres minéraux: carbominérite, chlorite, hématite, baryte, anhydrite, plagioclase, halite, rutile, kyanite, monazite, chalcocite, antlérite, serpentine.

4. Le tout-venant des mines de charbon du bassin de Sidney contient des inclusions minérales d'illite, de quartz, de kaolinite et de pyrite de la dimension des poussières inhalables. De plus, les roches provenant du toit ou roches clastiques syngénétiques produisent des grains détritiques de calcite, d'ankérite, de monazite, de zircon, de biotite, de plagioclase et de quartz. On trouve de nombreux minéraux accessoires syngénétiques ou épigénétiques, notamment du rutile et des produits d'oxydation de la pyrite qui constituent une grande partie des éléments traces.

5. Le poussier utilisé dans les mines de la SDCB est surtout composé de calcite mais contient également des quantités appréciables de baryte, sidérite, ilménite, colophane, célestite, pyrite, zircon, sphalérite et d'autres minéraux exotiques.

6. L'analyse par balayage et par points de particules de poussière de charbon par la méthode EDX, révèle la présence des éléments suivants:

   Principaux éléments: Si, Al, K, S, Fe, Ca

   Éléments mineurs ou traces: Cu, Ti, Cl, Mg, Mn, Na, Ba, P, Ce, Zn.

7. On a fait la découverte imprévue d'une substance riche en cuivre sur plusieurs filtres, dont le rapport de Cu au S laisse supposer qu'il s'agirait d'antlérite: Cu3(OH)4.SO4
   ou de
   chalcocite: Cu2S

   Cette substance s'est peut-être formée au cours du traitement ou de l'extraction du minerai.

8. La poussière de charbon recueillie au front de la longue taille comporte des concentrations en carbone et en azote (69 % de C; 1,55 % de N) quelque peu supérieures au tout-venant en général (61,7 % de C; 1,07 % de N). Ailleurs, l'analyse des éléments organiques contenus dans la poussière et dans le charbon d'origine indique les mêmes différences en teneur.

9. Les autres techniques d'analyse minéralogique et chimique appliquées à la poussière de charbon comportent les limites suivantes:

   Procédés minéralogiques

Diffraction des rayons X:	À cause des limites de détection et de l'interférence des valeurs maximales du spectre, cette technique ne convient pas à l'analyse de la poussière de charbon. Elle se limite à l'analyse semi-quantitative du quartz. De plus, son application à l'analyse des cendres formées à basse température n'est pas clairement établie.
IR	À cause de ses limites de détection, du brouillage à l'intérieur de la bande et de la préparation qu'elle nécessite, cette technique ne convient pas aux analyses, à l'exception du quartz qui est contenu dans la poussière de charbon.
Microscopie électronique par transmission	Convient à l'analyse de submicrons, mais non à l'analyse en vrac.
SEM-WDS	Les procédés optiques ne conviennent pas
Microsonde électronique	Les procédés optiques ne conviennent pas.

Procédés chimiques

INAA	Convient à l'analyse globale de la poussière de charbon en ce qui concerne 43 éléments, mais le délai d'exécution est excessif.
SSMS	Convient à l'analyse des éléments traces et des composantes organiques mais n'a pas encore été utilisé au Canada.

10. La microscopie électronique à balayage est utilisée pour l'analyse des poussières inhalables des mines de charbon depuis 1971 et fait l'objet d'un intérêt accru depuis la mise au point du dispositif de réglage automatique du faisceau et d'analyse d'images (AIA). Pour atteindre un niveau d'incertitude inférieur à 10 % dans l'analyse des macroparticules de poussière de charbon captées par les filtres, il faut se fonder sur plus de 10 000 points de données. Le recours à l'AIA et au SEM-EDX pour contrôler l'analyse, particule par particule, est nécessaire dans ce cas.

11. Il n'existe pas de normes appropriées à la micro-analyse par SEM-EDX des poussières de charbon inhalables.

12. Les filtres Nuclepore de polycarbonate sont les supports les plus appropriés à la microanalyse par SEM-EDX de la poussière captée. Les filtres Millipore d'acétate de cellulose peuvent être utilisés pour l'analyse manuelle par SEM-EDX, mais ne peuvent servir à l'analyse automatique des images. Les autres types de filtres (fibre de verre PVC, etc.) ne sont pas appropriés.

13. Les conditions optimales pour l'analyse manuelle des macroparticules de poussière de charbon au moyen du SEM-EDX sont les suivantes:

    a. filtre Nuclepore
    b. enrobage de carbone
    c. accélération de la tension du faisceau jusqu'à 20 kv.
    d. production d'images par rétrodiffusion des électrons (BEI)
    e. grossissement des éléments de 2 500 à 3 500 fois pour la production d'images ou de 10 000 fois pour l'analyse par point en mode SEI.

14. Les applications du logiciel commercial d'analyse quantitative par le procédé EDX, lequel utilise les corrections de matrices ZAF, sont limitées en ce qui concerne l'analyse des macroparticules de poussière de charbon, en raison des effets de la géométrie du faisceau et de l'absence de normes.

15. L'analyse par EDX, qui consiste à balayer les charges de poussière captées sur filtres, donne des comparaisons qualitatives qui laissent supposer que les tendances chimiques dépendent des postes de travail. Les renseignements obtenus sont, cependant, brouillés par des corrections de matrices ZAF inappropriées et par une précision médiocre.

16. L'analyse par EDX de la cendre de charbon fondue pour déterminer les principaux éléments peut s'appliquer à l'analyse des poussières inhalables et est compatible avec le logiciel pour analyse quantitative. Cependant, il faudra néanmoins en accroître la précision et optimiser la méthodologie de fusion du verre.

17. L'incinération à très basse température (plasma) de la poussière de charbon crée de nouvelles structures de macroparticules ainsi que des altérations chimiques. C'est pourquoi ce procédé ne convient pas à l'analyse minéralogique quantitative bien qu'elle puisse s'appliquer à la préparation des minéraux pour l'INAA ou à la concentration des minéraux accessoires.

18. Pour mettre au point une technique d'analyse minéralogique et chimique, d'exécution rapide, des poussières de charbon, il faudra perfectionner les procédés SEM-EDX et AIA et les méthodes de préparation des échantillons.

SUMMARY

1. The Atlantic Coal Institute has conducted a comprehensive investigation into analytical methods for mineralogical and multielemental chemical analysis of respirable coal mine dust. The most appropriate single analytical method for mineralogical, morphological, and chemical characterization of respirable coal dust is scanning electron microscopy with energy dispersive xray microanalysis (SEM-EDX) applied to "as captured" dust particles on filters.

2. Respirable coal mine dust from CBDC Mines collected on Nuclepore or Millipore filters showed the following average major mineralogy based on 500 point counts on six filters:

	wt%
coal	41.94
illite	31.94
calcite	3.88
quartz	9.49
kaolinite	2.29
pyrite	2.54
siderite	2.14
other	5.78
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3. Based on 3075 particulate analyses by SEM-EDX, the following additional minerals were identified or proposed: carbominerite, chlorite, hematite, barite, anhydrite, plagioclase, halite, rutile, kyanite, monazite, chalcocite, antlerite, serpentine.

4. Run-of-mine coal from Sydney Basin coal mines contain respirable dust size mineral inclusions of illite, quartz, kaolinite, and pyrite. In addition, roof rock or syngenetic clastics contribute detrital grains of calcite, ankerite, monazite, zircons, biotite, plagioclase, and quartz. Numerous syngenetic or epigenetic accessory minerals are present, including rutile, oxidation products of pyrite, etc., accounting for much of the trace element chemistry.

5. Stone dust used in CBDC mines is dominantly calcite but contains appreciable quantities of: barite, siderite, ilmenite, collophane, celestite, pyrite, zircon, sphalerite, and other exotic mineralogy.

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6. Area rasters and particulate spot analyses by EDX reveal the presence of the following elements in coal dust particles:

   major elements: Si, Al, K, S, Fe, Ca

   minor/trace elements: Cu, Ti, Cl, Mg, Mn, Na, Ba, P, Ce, Zn.

7. An unexpected discovery is the presence on several filters of a copper rich material tentatively identified by Cu:S ratios as: antlerite: $Cu_3(OH)_4.SO_4$
   or
   chalcocite: Cu_2S
   Derivation from the mining process or collection procedure is suspected.

8. Coal dust collected at the longwall face carries slightly higher carbon and nitrogen concentrations (69% C; 1.55%N) than the average R.O.M. coal (61.7%C; 1.07%N). Otherwise, organic elemental analysis on dust and parent coal reflect the sample heterogeneities.

9. Other techniques for mineralogical and chemical analysis applied to coal dust have the following limitations:

Mineralogical

XRD	Detection limits and peak interference unsuitable for coal dust. Limited to semiquantitative analysis for quartz. Application to low temperature ash not fully established.
I.R.	Detection limits, band interference and preparation requirements unsuitable except for quartz content in coal dust.
T.E.M.	Limited to submicron particles, unsuitable for bulk analysis.
SEM-WDS	Optics not suitable
EMP	Optics not suitable

Chemical

INAA	Suitable for whole coal dust analysis for 43 elements but with excessive turn-around.
SSMS	Suitable for trace elements and organic constituents but untested in Canada to date.

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10. Scanning Electron Microscopy has been applied to respirable coal mine dust since 1971 and with renewed interest since the development of automated beam control and image analysis (AIA). To achieve <10% uncertainty in particulate analysis of coal dust on filters requires >10,000 data points. This requires AIA with the SEM-EDX to control particle-by-particle analysis.

11. No appropriate standards exist for SEM-EDX microanalysis of respirable coal dust.

12. Nuclepore polycarbonate filters are the most appropriate substrate for "as captured" dust SEM-EDX microanalysis. Millipore cellulose acetate filters are usable for manual SEM-EDX but not suitable for automated image analysis. Other filter types (glass fibre, PVC, etc.) are unsuitable.

13. The optimum conditions for coal dust particulate analysis by SEM-EDX in manual mode are:

    a. Nuclepore filter substrate
    b. Carbon coating.
    c. Accelerating beam voltage of 20kv.
    d. Backscattered Electron Imaging (BEI).
    e. Magnification of x2500 to x3500 for imaging or x10,000 for point counts in SEI mode.

14. Commercial quantitative analysis software for EDX using ZAF matrix corrections have limited applications to coal dust particulate analysis because of beam geometry effects and the lack of standards.

15. EDX analysis by beam rastering over filter dust loadings provides qualitative comparisons that suggest chemical trends related to work stations. Information is, however, masked by inappropriate ZAF matrix corrections and poor sensitivity.

16. EDX analysis of fused coal ash for major elements has application to respirable dusts and is compatible with quantitative analysis software but requires further work to increase sensitivity and optimize glass fusion methodology.

17. Low temperature plasma ashing of coal mine dust creates particulate artifacts and chemical alterations. It is not appropriate for quantitative mineralogical analysis but may have application to INAA preparation or accessory mineral concentration.

18. The develoornent of a rapid routine mineralogical/chemical technique for coal mine dust requires further work in SEM-EDX-AIA and sample preparation procedures.

RECOMMENDATIONS

The following areas of further investigation are offered:

1. Sample collection procedures to standardize filter loadings and remove particulate distribution bias.
2. Sample preparation procedures of dust removal from Millipore and redeposition onto Nuclepore for optimum substrate.
3. Development of BEI threshold procedures for coal dust to allow application of automated beam control and image analysis.
4. Improvement of statistical data control on point count mineralogical analysis. Data counts must be increased to 10,000 by automation. Replicate collections and replicate analyses at high data density are required to develop confidence limits. Image analysis of dust particle mass is required for size distribution and weight % calculations.
5. Standards suitable for microanalysis and applicable to coal dust must be developed or identified.
6. At present, there is no facility in Canada that can analyse respirable coal mine dust mineralogy with confidence. The current study is a major advance in that direction. Nevertheless, all results to date must be treated with caution and not applied to regulatory requirements.

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INTRODUCTION

PROJECT CONCEPTS:

The incidence of pneumoconiosis in coal miners has been attributed to various constituents in respirable coal dust including the silica, trace elements, and organic compounds. Present practice is to monitor the weight of total respirable dust, the ash and free silica content, in relation to volume of air and exposure of workers.

Labour Canada has adopted the U.K. standards for Cape Breton coal mines which require that lifetime dust exposure of coal face workers average less than the following (Elfstrom (1975), see Appendix 1):

4.3 mg/m3 respirable dust
2 mg/m3 ash
0.15 mg/m3 silica

A 4.3 mg/m3 average dust level has been calculated to give a 3.4% maximum probability of contracting simple pneumoconiosis after 35 years of exposure. Such dust levels are monitored directly by personal samplers or indirectly by measurements of the mass concentration of respirable dust in the return airway.

The Canadian Centre for Mineral and Energy Technology (CANMET) has an ongoing research and analytical programme involving respirable dust. Their Elliot Lake Laboratory is equipped to calibrate dust detection instruments via a "respirable dust chamber" or to measure silica in dust via x-ray diffraction. In fact, the Labour Canada standards (Appendix 1) specify that

> "respirable quartz content of samples shall until further notice be determined by the Elliot Lake Laboratory of EMR using x-ray diffraction..."

Likewise, the Ontario Ministry of Labour, Occupational Health and Safety Division, by Ontario Regulation 769/83 have specified that alpha quartz on air sampling filters should be analysed by x-ray diffraction.

These XRD techniques were developed primarily for metal mines and have several limitations when applied to coal dust. Furthermore, the analytical results are limited to quartz content and do not address the range of mineralogy or trace elements that are encountered.

To reduce the risk of fire, the Canada Labour Code (Appendix I2) requires that every roadway in a coal mine be treated with "stone dust" to maintain a ratio of 75/25 with combustible coal dust. Such "stone dust" must consist of 70% by weight fines of -200 mesh. This stone dust can, therefore, make a substantial contribution to the respirable dust levels for certain work stations. The regulatory assessment of airborne dust by Labour Canada standards specifies the following:

1. Total Respirable Dust by gravimetric weighing to an accuracy of 0.03 mg.

2. Ash by ashing and weighing
3. Respirable Quartz by XRD or alternate approved technique

In order to assess airborne respirable dust as a factor in pneumoconiosis, the mineralogical and chemical composition of coal, airborne dust, and stone dust should be analysed more comprehensively than the regulatory requirements.

To assess the requirements for such a comprehensive analytical programme, we must consider the complex nature of the material in question. Gluskoter et al (1981) published a list of minerals that have been identified in coal or as oxidation products (Table 1). These minerals account for some of the long list of major, minor, and trace elements reported by analysts. Finkelman (1981) has tabulated the likely modes of occurrence of various elements either within the coal macerals, or mineral inclusions (Table 2). The relative contributions of these minerals and trace elements to respirable dust will likely vary with coalfield, workplace, and sampling procedure.

The request for proposal (07SQ-23440-4-9093, June 11, 1984) identified the following analytical objectives:

1. Mineralogical and elemental composition of mine dust.
2. Quantitative analyses of constituents in airborne respirable dust, total dust, stone dust, coal.

Modifications to these objectives on August 2, 1984 identified the requirement for:

3. Percentages of quartz, coal, stone dust, and trace elements in respirable airborne dust.

TABLE 1 Gluskoter _et_ _al_.,(1981)

Minerals Identified with Coals

Mineral	Formula
Clay minerals	
Montmorillonite	$Al_2Si_4O_{10}(OH)_2 \cdot H_2O$
Illite-sericite	$KAl_2(AlSi_3O_{10})(OH)_2$
Kaolinite	$Al_4Si_4O_{10}(OH)_8$
Halloysite	$Al_4Si_4O_{10}(OH)_8$
Chlorite (prochlorite, penninite)	$Mg_5Al(AlSi_3O_{10})(OH)_8$
Mixed-layer clay minerals	
Sulfide minerals	
Pyrite	FeS_2
Marcasite	FeS_2
Sphalerite	ZnS
Galena	PbS
Chalcopyrite	$CuFeS_2$
Pyrrhotite	$Fe_{1-x}S$
Arsenopyrite	FeAsS
Millerite	NiS
Carbonate minerals	
Calcite	$CaCO_3$
Dolomite	$(Ca, Mg)CO_3$
Siderite	$FeCO_3$
Ankerite (Ferroan dolomite)	$(Ca, Fe, Mg)CO_3$
Witherite	$BaCO_3$
Sulfate minerals	
Barite	$BaSO_4$
Gypsum	$CaSO_4 \cdot 2H_2O$
Anhydrite	$CaSO_4$
Bassanite	$CaSO_4 \cdot 1/2H_2O$
Jarosite	$(Na, K)Fe_3(SO_4)_2(OH)_6$
Szomolnokite	$FeSO_4 \cdot H_2O$
Rozenite	$FeSO_4 \cdot 4H_2O$
Melanterite	$FeSO_4 \cdot 7H_2O$
Coquimbite	$Fe_2(SO)_3 \cdot 9H_2O$
Roemerite	$FeSO_4 \cdot Fe_2(SO_4)_3 \cdot 12H_2O$
Mirabilite	$Na_2SO_4 \cdot 10H_2O$
Kieserite	$MgSO_4 \cdot H_2O$
Sideronatrite	$2Na_2O \cdot Fe_2O_3 \cdot 4SO_3 \cdot 7H_2O$
Chloride minerals	
Halite	NaCl
Sylvite	KCl
Bischofite	$MgCl_2 \cdot 6H_2O$
Silicate minerals	
Quartz	SiO_2
Biotite	$K(Mg, Fe)_3(AlSi_3O_{10})(OH)_2$
Zircon	$ZrSiO_4$
Tourmaline	$Na(Mg, Fe)_3Al_6(BO_3)_3(Si_6O_{18})(OH)_4$
Garnet	$(Fe, Ca, Mg)_3(Al, Fe)_2(SiO_4)_3$
Kyanite	Al_2SiO_5
Staurolite	$Al_4FeSi_2O_{10}(OH)_2$
Epidote	$Ca_2(Al, Fe)_3Si_3O_{12}(OH)$
Albite	$NaAlSi_3O_8$
Sanidine	$KAlSi_3O_8$
Orthoclase	$KAlSi_3O_8$
Augite	$Ca(Mg, Fe, Al)(Al, Si)_2O_6$
Hornblende	$NaCa_2(Mg, Fe, Al)_5(SiAl)_8O_{22}(OH)_2$
Topaz	$Al_2SiO_4(OH, F)_2$
Oxide and hydroxide minerals	
Hematite	Fe_2O_3
Magnetite	Fe_3O_4
Rutile	TiO_2
Limonite	$FeO \cdot OH \cdot nH_2O$
Goethite	$FeO \cdot OH$
Lepidocrocite	$FeO \cdot OH$
Diaspore	$AlO \cdot OH$
Phosphate minerals	
Apatite (fluorapatite)	$Ca_5(PO_4)_3(F, Cl, OH)$

Table 2. Suggested Modes of Occurrence

(From Finkelman, 1981)

Sb - probably as an accessory sulfide in the organic matrix
As - solid solution in pyrite
Ba - barite, crandallites, and other Ba-bearing minerals
Be - organic association, clay
Bi - accessory sulfide, perhaps bismuthinite
B - generally organic association, illite
Br - organic association
Cd - sphalerite
Cs - inorganic association, clays, feldspars, or micas
Cl - organic association, NaCl
Cr - clays
Co - associated with sulfides, such as pyrite and linnaeite
Cu - chalcopyrite
F - unclear, probably several inorganic associations, such as apatite, amphibole, clays, and mica
Ga - clays, organic association, sulfides
Ge - organic association, rarely in silicates, sphalerite
Au - native gold, gold tellurides
Hf - zircon
In - sulfides or carbonates
I - organic association
Pb - pyrite, galena, PbSe, coprecipitated with Ba
Li - clays - illite, mixed-layer
Mn - siderite, calcite
Hg - solid solution with pyrite
Mo - unclear, probably with sulfides, or organic constituents
Ni - unclear, may be with sulfides, organic constituents, or clays
Nb - oxides
P - various phosphates, some may be organically associated
Pt - perhaps native Pt alloys
REE- phosphates
Re - sulfides or organic constituents
Rb - probably illite
Sc - unclear, clays, phosphates or may have organic association
Se - organic association, as PbSe in Appalachian coals, pyrite
Ag - probably silver sulfides, but may occur in several modes
Sr - carbonates, phosphates, organic association in low rank coals
Ta - oxides
Te - unclear
Tl - sulfides, probably epigenetic pyrite
Th - REE phosphates
Sn - inorganic, tin oxides or sulfides
Ti - titanium oxides, organic association, clays
W - unclear, may have organic association
U - organic association, zircon
V - clays (illite)
Y - REE phosphates
Zn - sphalerite
Zr - zircon

PROJECT HISTORY

The present work resulted from our response to a request for proposal received on June 19, 1984, from the Department of Supply and Services (P.A. Mortimer, June 11, 1984, (File 7SQ-23440-4-9093). Our initial proposal, based on a start date of July 1, 1984, was revised on August 10, 1984, with expanded scope and a start date of September 1, 1984. The final contract (Table 3) was received on October 31, 1984, but activated by telex for October 11, 1984, with a scheduled completion date of March 1, 1984.

As indicated in the initial proposals, the work plan was dependent on the nature of samples to be collected by CANMET personnel. The majority of filter samples (68) were not received until February 18, 1985; therefore, the report deadline was revised to March 31, 1985.

The time factor was an important limitation to interlaboratory technique comparison as well as the trace element survey. Samples for neutron activation were transmitted to the subcontractor on January 21, 1985, and February 20, 1985; but because of the comprehensive suite of elements to be tested, results were not received in time for the body of this report and are included as appendix II-10. Likewise, evaluation of image analysis and beam control techniques of SEM-EDX at Tracor Northern (Middleton, Wisconsin) were not available but are included in appendix II-9.

Limitations to achieving the objectives initially proposed were also imposed by the nature of samples:

a. Quality of available standards and accuracy of their reported composition.

b. Concentrations of minerals and elements relative to analytical detection limits.

c. Limited dust sample size for comparative tests.

d. Filter types and loadings.

In view of these time and technical constraints, the project was fast-tracked by making contact with other research groups and published literature. The senior author met with staff of the Institute in Materials Science at the State University of New York (New Paltz) on October 15-19, 1984, to discuss particulate analysis by SEM-EDX. Subsequently, on October 22 he visited the Ontario Research Foundation, Materials Division, (Mississauga, Ontario) to see the SEM-EDX system used by Dr. E.J. Chatfield for asbestos particulate analysis. Becqueral Laboratories Inc. (Mississauga) and Dr. D.C. Stuart were visited to discuss procedures of neutron activation analysis.

On November 19-21, the Halifax laboratories of the National Research Council, Dalhousie University Department of Geology, and Department of Defence (Dockyard Laboratories) were contacted for purposes of literature and analytical procedure surveys.

Contact was also made with Dr. J. Mutmansky (The Pennsylvania State University), and the Elliot Lake Laboratory of CANMET at the suggestion of the Scientific Authority.

Further literature searches were conducted in Calgary, Alberta at the University of Calgary and the Institute of Sedimentary and Petroleum Geology. Visitations included the laboratories of Birtley Coal and Mineral Testing.

This survey has revealed that very little work has been done in Canada on coal fines or mine dust beyond regulatory measurements of dust volume and quartz content. Quantitative particulate analysis is an ongoing research concern of a number of North American research teams with limited success due to inherent problems of sampling, particulate characteristics, and analytical limitations.

Table 3.

Supply and Services Canada   Approvisionnements et Services Canada

CAU: --:04

11 LAURIER ST., HULL, QUEBEC K1A 0S5
11, RUE LAURIER, HULL (QUÉBEC) K1A 0S5

Atlantic Coal Institute
P.O. Box 1594
Sydney, Nova Scotia
B1P 6R8

24K96

P.I. – I.P. PAGE 1 OF/DE 6
DSS FILE NUMBER – NUMÉRO DU DOSSIER MAS: 07SQ.23440-4-9093
ADDRESS ENQUIRIES TO – ADRESSER LES DEMANDES DE RENSEIGNEMENTS À: P.A. MORTIMER (07SQ)
TEL.: (819/613) 997-7400

LOC: EEEE
TF: 03

CONTRACT - CONTRAT

YOU ARE REQUESTED to sell and/or supply to Her Majesty the Queen in right of Canada upon the terms and conditions set out herein and on the reverse side hereof, the supplies and/or services listed below and any attached sheets at the price(s) set out therefor.

NOUS VOUS DEMANDONS de vendre et(ou) de fournir à Sa Majesté la Reine du chef du Canada, aux conditions énoncées au recto et au verso de la présente et au(x) prix indiqué(s) les articles et(ou) les services énumérés ci-dessous et sur toute feuille ci-annexée.

f.o.b. POINT – POINT DE f. à b.: OTTAWA, ONTARIO
CONSIGNEE – CONSIGNATAIRE: Dept. Energy, Mines & Resources
CANMET, Coal Research Laboratories
555 Booth Street
CODE 23440 Ottawa, Ontario K1A 0G1

INSPECTION: ☒ CONSIGNEE – CONSIGNATAIRE ☐ DSS – MAS ☐ DND/QAD – MDN/DAQ

REQUISITIONING DEPARTMENT – MINISTÈRE DEMANDEUR: ENERGY, MINES & RESOURCES 23

ITEM NO. – Nº DE L'ARTICLE	NSN – NIBS	DESCRIPTION	U of I – U M D	QUANTITY – QUANTITÉ	UNIT PRICE – PRIX UNITAIRE
	Z-0001-N-R-J				

TITLE:

DEVELOPMENT AND DEMONSTRATION OF AN ANALYTICAL METHODOLOGY FOR ROUTINE DETERMINATION OF THE MINERALOGICAL AND ELEMENTAL COMPOSITION OF AIRBORNE DUST FROM UNDERGROUND COAL MINES

EFFECTIVE CONTRACT COMMENCEMENT DATE: October 11, 1984

STATEMENT OF WORK

You shall perform the work in accordance with the technical portion of your proposal dated July 3, 1984, your file No. 84-129 as amended by your letter dated August 10, 1984.

PRECEDENCE OF DOCUMENTS

In the event of conflict the precedence of documents shall be as follows:
1. this contract; 2. your proposal, File Ref. 84-129 dated July 3, 1984 as amended by your letter dated August 10, 1984.

CONSIGNEE – CONSIGNATAIRE	VALUE – VALEUR	CONSIGNEE – CONSIGNATAIRE	VALUE – VALEUR	COUNTRY OF ORIGIN – PAYS D'ORIGINE: CDN.	VALUE OF FOREIGN CONTENT – VALEUR ÉLÉMENTS ÉTRANGERS
CONSIGNEE – CONSIGNATAIRE	VALUE – VALEUR	CONSIGNEE – CONSIGNATAIRE	VALUE – VALEUR	REVENUE SHARING – PARTAGE DES RECETTES	

DELIVERY – LIVRAISON: MARCH 1, 1985
FROM DATE OF AWARD OF CONTRACT – À PARTIR DE LA DATE D'ADJUDICATION DU CONTRAT
TELEPHONE
DATE
FEDERAL SALES TAX – TAXE DE VENTE FÉDÉRALE: INCLUDED

We hereby AGREE to sell and/or supply to Her Majesty the Queen in right of Canada, upon the terms and conditions set out herein and on the reverse side hereof, the supplies and/or services listed above and on any attached sheets at the price(s) set out therefor.

Nous CONSENTONS de vendre et(ou) de fournir à Sa Majesté la Reine du chef du Canada, aux conditions énoncées au recto et au verso de la présente et au(x) prix indiqué(s) les articles et(ou) les services énumérés ci-dessus et sur toute feuille ci-annexée.

PRINT NAME – NOM EN MAJUSCULES

SIGNATURE

DISCOUNT TERMS – MODALITÉS D'ESCOMPTE: N/A

INVOICES ARE TO BE MADE OUT TO – ÉTABLIR LES FACTURES AU NOM DE

Send the ORIGINAL and ☒ copies to – Envoyer l'ORIGINAL et exemplaires au: See «Method of Payment» clause herein

☐ Send one copy to DSS – Envoyer un exemplaire au MAS

All invoices, shipping bills and packing slips must show the following numbers – Les factures, connaissements et inventaire du contenu doivent porter les numéros suivants

REQUISITION NO. AND FINANCIAL CODES – Nº DE LA DEMANDE ET CODES FINANCIERS:
RN: 23440-4-9093
FC: 214-131-000000-430350-0415

CONTRACT SERIAL NUMBER – NUMÉRO DE SÉRIE DU CONTRAT: 0SQ84-00240

CAP/PM – PAC/MEP: 07/08 $45,000.00
EST. EXPENDITURE – MONTANT EST.: $45,000.00
DATE: OCT 31 1984
VEP – LAS: B1P
FOR THE DEPARTMENT – POUR LE MINISTÈRE: P. A. Mortimer
SIGNATURE

DSS-MAS 1102E (10/83)

APPENDIX «A»

DSS File No.: 07SQ.23440-4-9093

TITLE: DEVELOPMENT AND DEMONSTRATION OF AN ANALYTICAL METHODOLOGY FOR ROUTINE DETERMINATION OF THE MINERALOGICAL AND ELEMENTAL COMPOSITION OF AIRBORNE DUST FROM UNDERGROUND COAL MINES

REQUIREMENT:

On behalf of the Department of Energy, Mines and Resources to complete by March 1, 1985 the development and demonstration of an analytical methodology for routine determination of the mineralogical and elemental composition of airborne dust from underground coal mines.

BACKGROUND:

The Cape Breton Coal Research Laboratory has initiated a long term research effort directed to the control of respirable airborne dust in Atlantic underground coal mines whose purpose is to prevent the development of coal workers' pneumoconiosis. While the specific etiological factors associated with this occupational disease have not been fully determined, the mineralogical and elemental composition of the airborne respirable dust is known to be one of the significant factors.

This contract is intended to develop and validate a routine analytical methodology that will be used by CBCRL researchers to determine the mineralogical and elemental composition of mine dust samples collected by gravimetric sampling. The technique may also be extended to samples collected by industry to meet regulatory requirements.

STATEMENT OF WORK:

1. Collect in consultation and with the approval of the Scientific Authority representative samples from CBDC underground mines of:
    a) airborne respirable dust
    b) airborne total dust
    c) stone dust
    d) coal from all seams worked

2. Perform either in-house or by subcontract detailed quantitative analyses of the samples collected in 1. Proposed analytical procedures must have the prior written approval of the Scientific Authority.

3. Determine in consultation and with the approval of the Scientific Authority, the constituents present in the samples collected in 1. that must be determined in the routine analytical methodology.

4. Using a range of standard reference materials, investigate various routine analytical techniques to quantitatively measure the constituents identified in 3.

5. Compare the various routine analytical techniques from 4. with emphasis on:
   a) accuracy,
   b) range,
   c) cost, and
   d) advantages/disadvantages of each technique.

6. Select the most appropriate candidate routine analytical methodology in consultation with the Scientific Authority.

7. Demonstrate the candidate analytical methodology with duplicate samples provided by the Scientific Authority by subjecting each sample to the candidate routine analytical metholology and the detailed quantitative analyses identified in 2.

8. Include all of the above in the final report to be provided to the Scientific Authority by March 1, 1985.

. . ./3

REPORTS:

The final report in ten (10) copies, is to be submitted at the conclusion of the work and this report must be comprehensive dealing with all facets of the work and must include sufficient drawings, sketches, photographs and discussions of problems and successes associated with the work to facilitate a full accurate evaluation of the work by the Scientific Authority. This report will be prepared in accordance with good engineering practice and will include as a minimum the following format: executive summary, introduction, discussion, conclusions and supporting graphs, tables and figures. The executive summary will be in both english and french. The final report shall be subject to inspection by the Scientific Authority prior to acceptance and should the report not be in accord with this contract, the Scientific Authority shall have the right to reject it or require its correction. Brief monthly reports in letter format shall be submitted to the Scientific Authority.

ACCEPTANCE:

All work is to be performed to the satisfaction of and is subject to acceptance by the Scientific Authority.

SCIENTIFIC AUTHORITY:

Dr. C.Y. Hwang
Cape Breton Coal Research Laboratory
CANMET
Department of Energy, Mines and Resources
210 George Street
Sydney, Nova Scotia
B1P 1J3

Telephone: (902) 539-5344

DEBRIEFING SESSION:

After submission and acceptance of the final report, the contractor shall prepare a verbal presentation outlining the problems and successes associated with the work and present this material in a debriefing session to CANMET representatives and interested outside parties. The time and location (usually Sydney or Ottawa) of the presentation will be at the discretion of the Scientific Authority who may, also, decide that the work does not warrant such a presentation. Costs associated with the debriefing in Sydney, N.S. are expected to be included in the bid.

CURRENT ESTIMATED COST: $45,000.

SAMPLE COLLECTION

a. AIRBORNE TOTAL/RESPIRABLE DUSTS

Collection of particulate samples for subsequent laboratory analysis can employ a variety of devices including:

1. High volume air samplers
2. Membrane filters
3. Continuous tape sampler
4. Cascade impactor
5. Electrostatic precipitator
6. Respirable dust sampler
7. Sedimentation collector

The Labour Canada Standards for dust in coal mines (Appendix 1) specifies a Cassella 113A gravimetric sampler for respirable dust. Samples received by our laboratory arrived as filter loadings obtained from personal samplers by staff of the Cape Breton Development Corporation or the Cape Breton Research Laboratory of CANMET. The later group supplied most of the samples based on their collection facilities including:

1. Dupont Model P2500A portable air sampling pump.
   From: Mine Safety Appliances Co. (Pittsburgh, Pennsylvania)
   This unit uses a selection of 25mm or 37mm filters made of cellulose, PVC or fibre glass. (See Figure 1).
2. Gravimetric dust sampler. Type 113A (National Coal Board).

The majority of samples made available to us by CANMET personnel (Table 4) were collected with a DuPont Model P-2500 constant flow personal sampler using a Casella gravimetric cyclone dust collector. This unit uses an external diaphram pump to sample at 1.9 liters per minute. Particles can be fractioned into respirable size, collected on a filter disc, and non-respirable particles collected in the grit pot.

There is considerable variation and uncertainty in the collection efficiency of such devices (Lippmann, 1978), with respect to flow rate, moisture effects, cyclone orientation, and correlation to pulmonary response. Furthermore, Foster and Walker (1984) have pointed out the need for carefull horizontal transport of filters back to the laboratory with minimum vibration. Such collection conditions were beyond our control and were likely difficult to standardize considering the logistics in C.B.D.C. coalmines. Several heavily loaded samples, for example, showed dislodged particle cakes. Other filters showed strong density zonation concentrated at filter centers.

BLE 4. COAL MINE DUST ON PERSONAL SAMPLER FILTERS: SYDNEY COALFIELD. Samples for mineralogical and elemental analysis**

ILTER TYPE	DESCRIPTION	COLLECTION PROCEDURES				NUMBER	SOURCE
		MSA Pump	Dupont Pump	Casella Cyclone	MSA Cyclone		
Millipore	mixed cellulose acetate and nitrate	x		x		7	CBCRL
	37mm dia.		x	x		56	CBCRL
	0.8 um pores	x		——open face——		3	CBCRL
uclepore	polycarbonate membrane	x		x		3	CBCRL
	37mm dia.	x			x	2	CBCRL
	0.8 um pores		x	x		5	CBCRL
	5.0 um pores	x		——open face——		1	CBCRL
	8.0 um pores	x		——open face——		1	CBCRL
MSA-FWS-B	polyvinyl chloride 37mm dia. 5.0 um pores	x			x	1	CBCRL
Glass	microfibre 55mm dia.	——Casella 113A Elutriator——				7	CBDC
					TOTAL:	86	

** Note: individual samples are identified in the Appendix I4.

MSA GRAVIMETRIC DUST SAMPLER
(Mine Safety Applicances Co.)

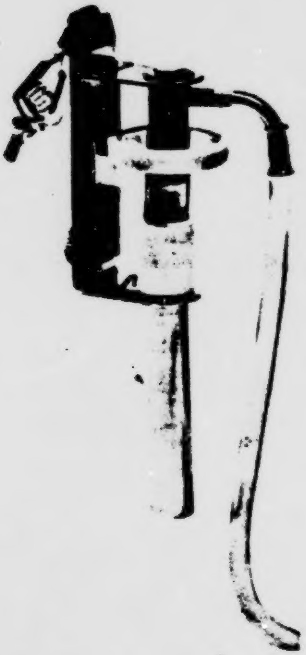


Figure 1.

DESCRIPTION

The MSA Gravimetric Dust Sampler is a personal sampler for the measurement of air-borne dust in industry. The unit consists of a 10 mm cyclone assembly and a pre-weighed cassette or filter holder.

OPERATION

A pump draws dust-laden air through the cyclone and cassette at a pre-selected flow rate. The sample enters the cyclone tangentially and the centrifugal motion of the flow increases the inertia of the larger, non-respirable particles such that they separate from the air stream and are collected in the base of the unit. The smaller, respirable particles remain in the air stream and are removed in the pre-weighted cassette. At the end of a suitable sampling period, the total weight of respirable dust in the cassette filter media is established and this figure plus the larger particles collected in the base gives the total dust concentration per cubic meter of air sampled.

SOURCE

Mine Safety Appliances Co., 400 Penn Centre Blvd., Pittsburgh, Pa. 15235.

b. PARENT COAL (R.O.M.)

Run-of-mine (R.O.M.) samples of approximately 100 kg were collected from five seams and locations in the Sydney Coalfield during the period of April to May 1984, as part of other research.

Each sample was stored in five airtight 6.5 gallon polyethylene Can-Am containers purged with nitrogen. By the ASTM standard D2234, these samples are of Type II increments, with random spacing under conditions D (stockpile) or A (underground belt). The moisture content was dependent on mine and weather conditions; and top size varied depending on the handling facilities.

Bulk sample preparation included air drying, hand crushing, and pulverizing using a Straub grinding Mill and Holmes Pulverizer to produce representative splits.

c. STONE DUST

A pulverized sample of stone dust used in the C.B.D.C. mines was supplied by CANMET personnel.

ANALYTICAL OBJECTIVES

a. MINERALS OF INTEREST

We previously identified some 57 minerals that have been reported from coals (Table 1). This list can be substantially reduced when considering significant contributions to respirable dust. Sprunk and O'Donnell (1942) studied some 3000 thin sections from 100 coal beds to identify the major minerals as:

A. Common Minerals (abundance order):

1. Kaolinite
2. Pyrite
3. Calcite
4. Quartz
5. Siderite

Gluskoter (1975) reviewed more recent work and added the following minerals to the list:

6. Illite
7. Montmorillonite
8. Ankerite
9. Dolomite
10. Iron sulphates from pyrite weathering

Dust size is measured in micro-meters or microns (1.0 um = 0.001 mm) based on "aerodynamic diameter" ie. a theoretical spherical particle of unit density that settles at the same speed as the irregular particle in question. Coal dust covers the particle range of 100 microns down to less than 0.01 microns, but the respirable dust range of interest is 0.1 to 10 microns. Dust with an aerodynamic diameter of about 7 um and above will not normally reach the alveoli and thus is not "respirable". Particles below 0.1 um in diameter are expelled during exhalation. Table 5. illustrates the relationship between particle size and coal dust character.

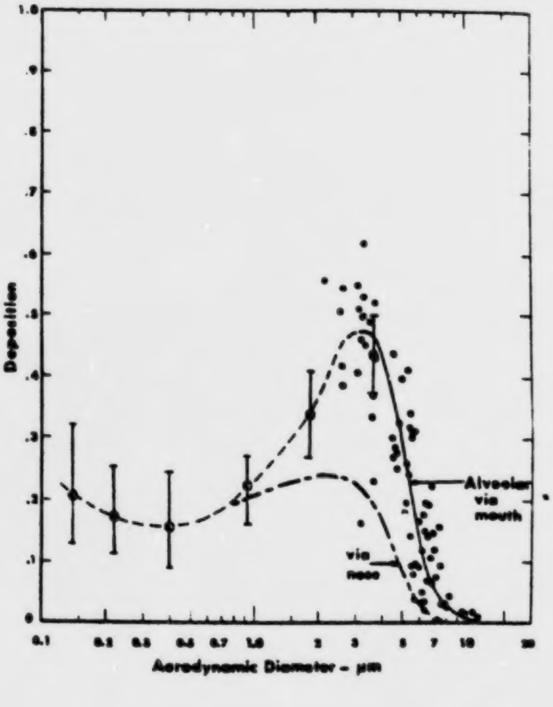
Table 5. **Relation Between Particle Size and Properties of Coal Dust**

Particle Size (microns)*	Time to Fall 3 feet	Hazard	Remarks
1	8 hours	(Health	(Respirable
		(	(
5	20 minutes	(	(
25	1 minute	(Explosion	(Dust can be made
		(	(airborne and ignited
100	4 seconds	(	(by a methane explo-
		(	(sion for example
500	Very short	None	
	*1 micron ( )	$\frac{1}{25\ 000}$ inch	

*Source: NCB Mining Research Establishment, Isleworth Middx, England, Bulletin 5, September 1964.

From: Appendix Report of Commission of Inquiry
Explosion in No. 26 Colliery, Glace Bay, Nova Scotia
February 24, 1979

Figure 2. Lippman (1978)



Deposition in the nonciliated alveolar region, in % of aerosol entering the mouthpiece as a function of aerodynamic diameter, except below 1/2 µm, where linear diameter was used.

. The dashed line is an eye-fit through the median best estimates of Altshuler on 3 subjects who varied as shown by the vertical lines. The lower curve is based on the additional head correction resulting from inhalation via the nose. (Reprinted courtesy The American Physiology Society).

It has been demonstrated by many investigators that mineral inclusions occur in coal macerals down to the limits of observation. Harris et al (1977) reported an average size range of 1 to 0.1 um but detected particles as small as 200°A. Finkelman and Gluskoter (1981) reported that most dispersed minerals in coal occur below the limits of resolution of optical microscopes and down to 0.04 um.

From the foregoing, we must conclude that at least 10 minerals may contribute to respirable dust in coal mines. Of these, quartz has attracted the most attention. Mutmansky and Lee (1984) report, however, that the effect of quartz on the incidence of pneumoconiosis is uncertain. Both the mass of dust and quartz content have positive correlations with coal workers' pneumoconiosis (CWP), but the effect of quartz is not felt until concentrations reach 10%. They conclude, therefore, that CWP is a multivariate problem.

b. ELEMENTS OF INTEREST

Table 1. gave the variety of chemical formulae represented in coal minerals. The major minerals indicate the major elements expected: Si, Al, K, Fe, S, Ca, Mg.

In addition, we must consider the major elements bound in the coal macerals including: C, H, N, O, and S and organic Si, Al, Ti, etc.

The variety of trace elements that can be encountered is listed in Table 2 but most will be at concentrations below detection in respirable dust samples. Elements that have been shown to accumulate in miners' lungs (Table 6) may not necessarily be in detectable concentrations in a single filter loading.

Table 7. lists a few trace element ranges encountered in Sydney coal samples for comparison against the similar list of elements from miners' lungs. (Table 6).

To the foregoing, we must add the anticipated contribution of stone dust to the respirable dust filter loadings. Depending on workplace, from 0 to 75% of airborne dust may be derived from the introduction of stone dust considering the 75:25 concentrations mandatory for roadways. Table 8. gives a typical range of analyses for stone dust as received from a supplier.

From the foregoing, it is evident that the number of elements contributing to respirable dust samples can be extensive. Mutmansky and Lee (1984) in a recent survey of Pennsylvanian

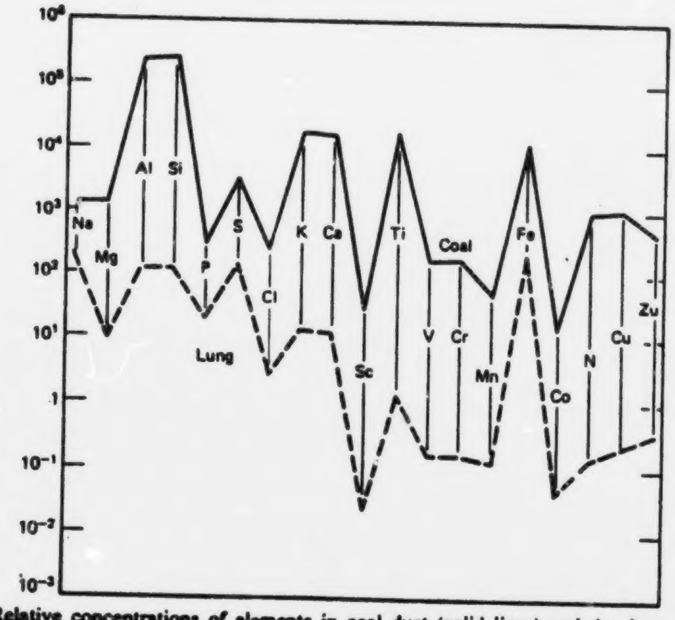
Table 6. *Comparison of Levels of Elements in Miners' Lungs with Normal Lungs*[a]

Element	Mean concentration (μg per dried lung) Miners' lungs (M)[b]	Normal lungs (N)[b]	Ratio (M/N)
Al	9535	205	47
Ba	283	2.5	113
Be	4.0	—	—
B	7.7	0.28	27.5
Cr	11.6	3.02	3.8
GE	1.1	—	—
Fe	1595	2410	0.66
Pb	23.7	13.9	1.7
Mg	98.8	495	0.20
Mn	8.1	4.38	1.8
Ni	37.0	20.2	1.9
Ag	1.1	0.01	110
Sn	20.0	23.13	0.87
Ti	215	50.1	4.3
V	45.2	1.73	26

[a] From Freedman and Sharkey (1972).

[b] Adapted from data of Crable *et al.* (1967).

Figure 3.



Relative concentrations of elements in coal dust (solid lines) and dry lung tissue

From: Sharkey and McCartney (1981)

TABLE 7.

MINOR ELEMENTS IN SYDNEY, NOVA SCOTIA COAL

	In coal, ppm Average	Range
Arsenic	100	33 - 270
Barium	34	2 - 257
Berylium	2	1 - 2
Boron	17	6 - 25
Chromium	5	2 - 9
Cobalt	10	3 - 34
Germanium	5	1 - 8
Lead	66	25 - 120
Manganese	140	9 - 254
Molybdenum	7	2 - 12
Nickel	15	6 - 74
Strontium	65	76 - 87
Tin	1	
Vanadium	14	7 - 28
Zinc	25	13 - 64

Table 8. Typical Analysis of Stone Dust
as Received from Supplier - November 20, 1974

Screen Analysis	Princess Colliery	No. 26 colliery	Lingan Colliery
	%	%	%
+ 28 Mesh	0.3	NIL	NIL
+ 50 Mesh	0.7	0.6	2.0
+ 100 Mesh	0.4	0.85	1.0
+ 200 Mesh	0.9	1.00	1.5
- 200 Mesh	97.7	97.55	95.5
Chemical Analysis			
H_2O	0.49	0.50	0.30
Ash	55.94	56.21	56.60
CO_2	43.40	43.06	42.16
Inert	99.83	99.77	99.06
Combustible	0.17	0.25	0.94
Loss on Ignition	42.28	42.56	42.40
SiO_2	2.96	2.09	2.90
* R_2O_3	1.96	1.12	1.12
CaO	51.65	51.32	51.32
MgO	1.60	1.90	1.00
MnO	0.21	0.21	0.21
** Sulpher	0.016	0.014	0.014

Source: Reports of Analyses conducted by Devco Laboratory, Victoria Junction, Coal Preparation Plant.

From: Appendix Report of Commission of Inquiry
Explosion in No. 26 Colliery, Glace Bay, Nova Scotia
February 24, 1979

* assume Fe_2O_3

** assume Sulphur

coalfields and coal workers' pneumoconiosis, concentrated on the following trace elements: Cr, Ni, Sr, Zr, Zn.

This listing was biased by the available Penn State Coal Data Bank and strong correlations with coal field and rank. A more comprehensive list of 15 elements were identified by Bombelka _et al_ (1984) to be of environmental and toxicological concern in a German aerosol project: S, V, Cr, Mn, Co, Ni, Cu, Zn, As, Se, Br, Cd, Sb, Hg, and Pb. The present study has emphasised the most comprehensive multielement analysis possible by INAA and EDX.

ANALYTICAL OPTIONS

INTRODUCTION

The comprehensive analysis of respirable coal dust for both mineral and elemental composition has never previously been accomplished in Canada. The factors that make it such a difficult task are:

1. Complex mineral suite
2. Amorphous organic matrix
3. Multielemental composition
4. Ultrafine particulate morphology
5. Small sample size
6. No standards

The selection of analytical options must, therefore, address a multivariant problem. Sharkey and McCartney (1981) have listed some techniques applicable to coal. Techniques that address mineralogy and chemistry are reviewed in the following pages, including methods not in Table 9.

Table 9.

Summary of Analytical Techniques for Coal

Technique	Information Derived
Optical properties	True rank criterion Size and ordering of aromatic layers Graphitic structure and anisotropy
Density and pore structure	Reactivity Diffusion of gases Absorbing power Molecular sieve material
Electron microscopy	Ultrafine granular structure Pore structure Graphitic crystallization and layering Element distribution in fine components
X-Ray diffraction	Size distribution of aromatic ring systems Diameter and thickness of lamellae Mean bond length
Electrical properties	Free radical concentrations Ring structure and sizes Energy gap–semiconductor properties Graphitic structure and anisotropy
Thermal analysis	Rate of change of physical state
Coal liquids—sample separations	Solvent separation Polarity Functionability
Mass spectrometry	Molecular weight and formulas Hydrocarbon type analysis Carbon number distribution Trace elements
Nuclear magnetic resonance	H_{ar}/H, C_{ar}/C ratios Distribution of isotopic tracers
Electron spin resonance	Free radicals, charge carriers
Infrared absorption	Functional groups (e.g., OH, CH_{ar}.CH_{al}, CH_3) Minerals
Ultraviolet-visible absorption	Aromaticity Aromatic ring size
Optical fluorescence	Identification of coal-derived components
Electron spectroscopy for chemical analysis (ESCA)	Elemental distribution and chemical state of surfaces
Emission spectroscopy	Quantitative and qualitative elemental analysis Single element and survey analysis Major, minor, and trace element analysis for metals and semimetals
X-Ray fluorescence	Inorganic element analysis
Atomic absorption	Precise quantitative determinations for metals and semimetals Single element analysis for minor and trace constituents
Mössbauer spectroscopy	Iron compounds and association with sulfur

From: Sharkey & McCartney (1981)

XRAY DIFFRACTION

a. QUALITATIVE

Xray diffraction is recognized as the best method for mineral identification in coal LTA (Jenkins and Walker, 1978). The Mineral Constitution Laboratories of Pennsylvania State University use -200 mesh low temperature ash (LTA) ground in alcohol and dispersed as slurry onto a glass slide. Xray diffraction profiles are based on monochromatic CuK radiation and scans of 2 - 50 (2θ). The principal xray diffraction spacings for common coal minerals are given in Table 10.

The above procedures are not sufficient to identify the different clays which may account for up to 60% of coal minerals. Hutchison (1974) outlined, in detail, the procedures for routine mineralogical analysis of powdered rock, including clay identification by glycolation and thermal treatment. Even such extensive sample processing is insufficient for unambiguous clay identification and Hutchison (1974) recommends supportive differential thermal analysis.

b. SEMI-QUANTITATIVE

Quantitative analysis by XRD is an order of magnitude more difficult . Hutchison (1974) presents a procedure for modal analysis of silicate rocks provided the following conditions are met:

Table 10. *Principal x-Ray Diffraction Spacings of Commonly Occurring Coal Minerals*[a]

Mineral	Diffraction spacing (Å)
Kaolinite	7.15(100), 3.57(80), 2.38(25)
Illite	10.1(100), 4.98(60), 3.32(100)
Montmorillonite	12.0–15.0(100)
Chlorite	14.3(100), 7.18(40), 4.79(60), 3.53(60)
Mixed layer illite–montmorillonite	10.0–14.0(100)
Calcite	3.04(100), 2.29(18), 2.10(18)
Dolomite	2.88(100), 2.19(30)
Siderite	3.59(60), 2.79(100), 2.35(50), 2.13(60)
Aragonite	3.40(100), 3.27(52), 1.98(65)
Pyrite	3.13(35), 2.71(85), 2.42(65), 2.21(50)
Marcasite	3.44(40), 2.71(100), 2.41(25), 2.32(25)
Quartz	4.26(35), 3.34(100), 1.82(17)
Gypsum	7.56(100), 4.27(50), 3.06(55)
Rutile	3.26(100), 2.49(41)
Feldspars	3.18–3.24(100)

[a] Relative intensities are shown in parentheses.

Figure 4.

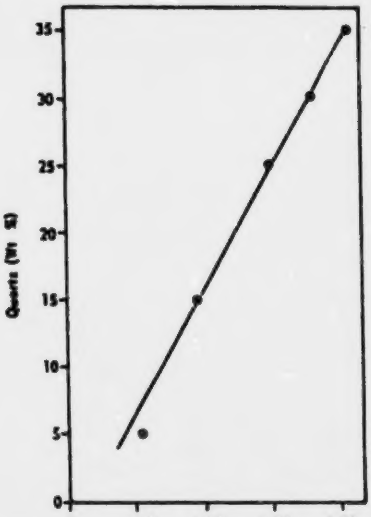


Fig. 1 X-ray calibration plot for quartz.

From: Jenkins and Walker (1978)

1. Particle size <5 um.
2. Mineral lines must not interfer.
3. Standards of pure minerals identical to constituents in the unknown.
4. No orientation of platy minerals allowed.
5. Only crystalline components present.

Under these conditions:

$$C = \frac{A_x I_x S}{A_s I_s}$$

where C = weight fractions of component in unknown
A_x = mass absorption coefficient of specimen (x)
I_x = intensity of diffraction
S = weight fraction of component in standard (s)

Jenkins and Walker (1978) report that quantitative analysis of coal LTA has been conducted for a limited number of minerals: pyrite, calcite, and quartz using an internal CaF_2 standard and calibration standards of mineral mixtures: calcite, pyrite, quartz, illite, kaolinite, and montmorillonite. Specimens are scanned at slow speed (0.25 - 0.50 (2θ/min) in a rotating sample mount. Peaks are measured for quartz (001), calcite (104), pyrite (311), and calcium fluoride (111) to establish calibration curves for each mineral (Figure 4).

This semiquantitative method has severe limitations for our purposes:

1. Sample size: 0.5g LTA ash
2. Pure mineral standards are required, while many coal minerals are solid solution series (eg. calcite-dolomite-ankerite)
3. Platey or fibrous minerals show preferred orientation, which hampers quantitative results.

4. Overlapping peaks: (eg. illite, pyrite)
5. Limited mineral suite: clays cannot be quantified.
6. The use of low temperature ash introduces up to 30% amorphous artifacts, (Kacsmar and Tomb, 1984) as well as oxidation products eg. bassinite $CaSO_4.H_2O$ (from organic sulphur), and Fe_2O_3 from pyrite, thereby distorting original concentrations.

Finkelman and Gluskoter (1981) conducted a round robin of LTA analysis by XRD and reported the following:

> "Recently, an interlaboratory study was designed to evaluate quantitative XRD results from different laboratories (Finkelman, Fiene, and Miller, unpublished data). The coal sample collected was ground, homogenized, and LT-ashed. The ash was blended, then distributed to the participating laboratories. Thirteen labs reported what they considered to be quantitative analyses on aliquots of the LT ash. The range for the major minerals varied from a factor of 2 (14 to 25 weight percent for quartz) to a factor of 4 (10 to 39 weight percent for pyrite).
>
> It seems the ability to quantitatively determine the minerals in an LT ash sample is still to be perfected."

c. QUARTZ BY XRAY DIFFRACTION

Xray diffraction procedures applied to respirable dust have generally been restricted to the analysis of quartz. Xray has advantages for use in metal mines because it can differentiate between polymorphs of free silica (crystobalite, tridymite, and quartz) which show differences in toxicity. Only quartz has been reported from coal mines.

Friedman (1978) reported on the U.S. Bureau of Mines procedure entitled:

> Standard Method 47: Determination of Quartz in Respirable Mine Dust on Membrane Filter by Xray Diffraction.

Mine dust samples are collected on an organic polymer membrane filter which is subsequently ashed, and inorganic components are redeposited onto a Gelman DM450 polyvinyl chloride acrylonitrile copolymer. Diffractograms are obtained using CuK radiation with a 0.008mm nickel filter.

Calibration is by a series of Novaculite quartz standard filter deposits prepared in isopropanol over the concentration range of 25-150mg/cm2.

Freeman (1978) points out that in xray analysis, the instrumental response is highly dependent on particle size and surface conditions; therefore, no single invariate standard exists for interlaboratory comparisons. Kacsmar and Tomb (1984) reported a 21% variation for five different quartz standards and 100mg loadings. The accuracy of this procedure is, therefore, unknown.

The Ontario Ministry of Labour, Occupational Health and Safety Division, has published specific XRD procedures for determination of alpha quartz on air sampling filters (Ontario Regulation 769/83, effective December 9, 1983). This technique is an on-filter analysis using 25mm silver membrane filters.

The air sampling is by a portable personal sampler with a filter assembly of 25mm silver membrane having a 5.0um pore size and a cellulose filter support. The large pore size is required for low resistance collection but results in the loss of much of the respirable dust (Freedman 1978, p. 323). At low concentration levels, the fine quartz is trapped in the silver pores and effectively screened from the xrays, reducing analytical sensitivity.

Xray diffraction is carried out with a cobalt target and a graphite monochromator using Ottawa silica sand (<10um) in epoxy as an external standard. Both the (101) and (112) peaks of quartz are monitored over the 20 ranges of 29.8°-31.9° and 58.2° to 59.7°

The 1983 regulations claim that over the range of 0.01mg to 2.00mg loadings on a 25mm filter, interlaboratory precisions are ± 0.02mg. Accuracy is not known and several interferences are anticipated (Freedman, 1978; Regulation 769/83):

1. Minerals that interfere with the (101) peak of quartz: biotite, muscovite, illite, microcline, plagioclase, graphite, kaolinite.
2. The presence of iron degrades peak resolution.

Freedman (1978) recommends the silver membrane procedure for non-coal mines where quartz concentrations are high. Alternatively, he mentions collection on a large organic membrane filter for ashing and subsequent redeposition to a 13mm silver membrane.

d. OUR STUDY

The foregoing literature survey indicates that XRD techniques are not appropriate for our objectives of complete mineralogical characterization of respirable coal mine dust. The sensitivity for individual minerals is too low for coal based dust and concentration by LTA is precluded by the small sample size. The complex mineral assemblages in coal will result in substantial peak interference precluding successful modal analysis, even on concentrates.

To illustrate the limitations, a set of Eastern Canadian Coals were prepared by low temperature ashing following modified procedures of Gluskoter (1965) described elsewhere. Samples of -60 mesh coal were ashed to completion in 9-11 days at 85 watt setting in an oxygen atmosphere. Under such conditions, we expect up to 30% of pyrite to convert to hematite and substantial amorphous artifacts (Miller et al 1979). Routine semi-quantitative XRD analysis was performed by a subcontractor (Xray Assay Laboratories Ltd., Don Mills, Ontario) using instrument procedures essentially set up for silica on silver membrane filters:

1. Powdered sample is hand ground in an agate mortar to -200 mesh without dilution.
2. Powder is mounted in a standard Philips holder and flattened with a glass sheet.

3. Xray diffractograms are obtained via a Philips 1710 unit operated at 40kv and 40ma using a cobalt tube. The scan covers 5-70° at a rate of .02/sec.
4. Mineral identification is manual via the JCDS file.

According to Hutchison (1974), a Co xray tube gives better peak resolution than CuK radiation and is especially preferred for minerals high in iron. Nevertheless, Kirkert and Brandt (1981) and Jenkins and Walker (1978) recommended CuK radiation for LTA from coals.

Table 11 reports the semi-quantitative mineral estimates for each coal LTA. For the major minerals: pyrite, quartz, and kaolinite in an 0.5 gram ash sample, only rough estimates are possible and even these are doubtful:

1. Pyrite is likely underestimated by 30% because of extensive oxidation to Fe2O3.
2. Quartz estimates suffer interference from kaolinite, illite and chlorite.
3. "Kaolinite" must be considered a group name for all the clays.
4. The concentration of quartz and "kaolinite" are likely overestimated. (Jenkins and Walker 1978, p 284).

For the minor mineral components the XRD results are obviously in error. Correlations with optical and SEM-EDX observations suggest that "gypsum" is likely an LTA artifact and

TABLE 11 X-RAY DIFFRACTION RESULTS OF LOW TEMPERATURE ASH[1]

MINE / SEAM	% MINERALS[2]						
	PYRITE	QUARTZ	KAOLINITE	GYPSUM	CALCITE	DOLOMITE	CHLORITE
LINGAN / HARBOUR	5–10	30–40	10–20	-	-	-	10–20
NOVACO / HARBOUR E	40–50	20–30	10–20	-	-	-	<5
BROGAN / LLOYD COVE	30–40	10–20	20	<5	-	-	ILLITE ?
EVANS / NO.5	30–40	20–30	5–10	-	-	5–10	<5
DRUMMOND / SCOTT	5–10	20–30	30–40	<5	-	-	<5
DRUMMOND / ACADIA	10–20	30–40	20–30	-	-	-	<5
MINTO / CHIPMAN 1	30–40	20–30	5–10	-	5–10	-	<5
MINTO / CHIPMAN 2	40–50	10–20	10–20	-	-	-	<5

1 Co tube, 2 theta scan: 5–70

2 as reported by subcontractor
Note: "kaolinite" is likely various clays
"gypsum" is likely LTA artifact
"chlorite" is likely illite

"chlorite" is more likely illite. Several minor components identified by SEM-EDX such as biotite, apatite, rutile, and siderite are not reported. The more common carbonate in these samples is ankerite and no dolomite has been found. If such poor results can be expected from bulk coals, the application to respirable dust is certainly questionable.

INFRARED SPECTROSCOPY

a. QUALITATIVE

The second most prevalent instrumental technique for characterizing minerals in coal is Infrared (IR) absorption spectroscopy. Identification of phases is by characteristic absorption bands due to atomic vibrations and rotations absorbing a primary infrared beam. Three levels of complexity are in use:

1. Standard double-beam dispersive I.R. spectrophotometer.
2. Computer interfaced differential scanning IR spectrophotometer allowing spectral enhancement, data averaging, etc.
3. Fourier transform I.R. spectrophotometer.

Dispersive instruments use prisms or gratings to disperse the infrared beam so that only a narrow frequency range falls on a set of slits. The prisms or gratings are rotated so that the intensity of the beam, modulated by the sample, is recorded as a function of frequency. High resolution spectra require narrow slits which attenuates the signal . FTIR, in contrast, uses an interferometer to allow the detector all the beam energy resulting in up to a 100 fold increase in signal. A Fourier transform is performed on the interferogram by minicomputer to yield total spectra within seconds. Multiple scans can be added to enhance the signal to noise ratio. For coals, more than 500 scans are needed to characterize constituents (Jenkins and Walker 1978). The analysis of a multicomponent mixture by FTIR is based on successive subtraction of spectra of individual components from the mixture spectrum. Although FTIR is a promising new tool for coal mineral analysis, it is not a routine procedure and, therefore, beyond the scope of the present survey.

Conventional dispersive I.R. spectroscopy shows similar technical limitations as XRD (Jenkins and Walker 1978):

1. The presence of a coal matrix causes broad bands overlapping constitutent mineral bands; therefore, LTA treatment is necessary.
2. Particle size is a critical factor and must be less than the wavelength of the light source.
3. Complex mineral mixtures suffer from band overlap, eg. pyrite overlaps with kaolinite.

Foster and Walker (1984) and Jenkins and Walker (1978) illustrated the significance of particle size and surface area (Figures 5a-d) and listed the interfering mineral bands, especially as apply to quartz (Table 13). Conventional I.R. spectroscopy has, therefore, been restricted to identification or semiquantitative analysis for selected minerals (kaolinite, gypsum, quartz). As for XRD, the technique tends to overestimate the contribution of kaolinite and quartz.

Table 12.

Infrared Absorption Bands for Standard Minerals

Mineral	Source	Absorption bands (cm⁻¹)
Kaolinite	Georgia	3695, 3665, 3650, 3620, 1108, 1025 1000, 910, 782, 749, 690, 530 460, 422, 360, 340, 268
Illite	Fithian, Illinois	3620, 1640, 1070, 1015, 920, 820 750, 510, 460
Sodium montmorillonite	Wyoming	3625, 3400, 1640, 1110, 1025, 915 835, 790, 515, 460
Calcite	Chihuahua, Mexico	1782, 1420, 871, 842, 710, 310
Dolomite	Lee, Mass.	1435, 875, 730, 390, 355, 310
Pyrite	Rico, Colorado	411, 391, 340, 284
Quartz	Minas Gerais, Brazil	1160, 1065, 790, 770, 687, 500 450, 388, 362, 256
Gypsum	Washington County, Utah	3605, 3550, 1615, 1150, 1110, 1090 1010, 660, 595, 450

From: Jenkins and Walker (1978)

Table 13.

Positions of the main absorption bands for minerals that commonly interfere in crystalline silica analysis

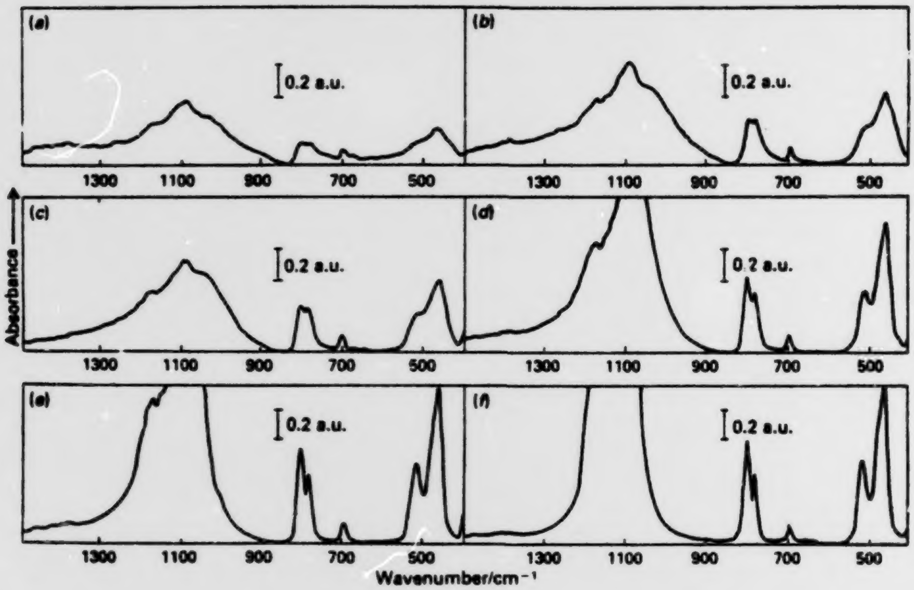
Mineral	Band wavelength*/cm⁻¹
Albite	402 (m), 418 (m,sh), 432 (ms), 465 (m,d), 477 (m,d), 535 (m), *598* (ms), 614 (m,sh), *652* (m), 726 (mw,q), 746 (m,q), 788 (m,q), 1005 (s,d), 1039 (s,d), 1105 (s,d), 1157 (s,d)
Anorthite	409 (m), 435 (m,sh), 481 (m), 538 (m), 577 (m), *625* (mw), *669* (mw), 728 (mw,d)), 758 (mw,d), 950 (s,d), 1021 (s,d), 1084 (ms, sh), 1160 (ms)
Cristobalite	496 (ms), *622* (w), *797* (mw), 1103 (s), 1199 (ms, sh)
Daphnite	429 (ms,sh), 467 (ms), 539 (m), 610 (m), *667* (m), 771 (m), 798 (m), 991 (s)
Kaolinite	433 (m), 474 (ms), 547 (ms), 695 (mw), *754* (w,d), 795 (w,d), *914* (m), *938* (m,sh), 1010 (s,d), 1033 (s,d), 1115 (m)
Marcasite	421 (mw), 533 (mw), 605 (mw,d), 625 (mw,d), *667* (mw), 829 (m), 1016 (m), 1081 (s), 1153 (s)
Mullite	468 (ms), *591* (s), 748 (s), 837 (s), 914 (s), 1135 (ms,d), 1162 (ms,d)
Muscovite	481 (ms), 535 (ms), *695* (w), 751 (w), 802 (w), *829* (w), 935 (m,sh), 1029 (s), 1062 (ms,sh)
Nepheline	432 (s), 475 (s,sh), 512 (m,d), 542 (m,d), *593* (ms), *645* (m), *719* (ms), 762 (m,sh), 1014 (s), 1132 (s)
Orthoclase	435 (ms), 464 (mw,sh), 540 (mw), 593 (m), *649* (mw), 728 (mw,t), 745 (mw,t), 767 (mw,t) 788 (w,sh), 1046 (s), 1140 (s)
Quartz	467 (ms), 524 (mw), *695* (w), *780* (m,d), 799 (m,d), 1087 (s), 1164 (ms,sh)
Ripidolite	482 (ms), 571 (m), 647 (mw,d), 669 (mw,d) 765 (mw), *880* (m,d), *904* (ms,d), 992 (s)
Steatite	426 (ms,sh), 451 (s,d), 467 (s,d), *670* (ms), 761 (w), 826 (w), 960 (ms, sh), 1016 (s)
Tridymite	479 (ms), *568* (w), 789 (m), 1058 (s,d), 1110 (s,d)

* w = weak (<20% A) (A = absorption); mw = medium weak (20–40% A); m = medium (40–60% A); ms = medium strong (60–80% A); s = strong (>80% A); sh = shoulder; d = doublet; t = triplet; q = quadruplet. Bands in italics are used for scaled absorbance subtraction.

From: Foster and Walker (1984)

Figures: a, b, c, d

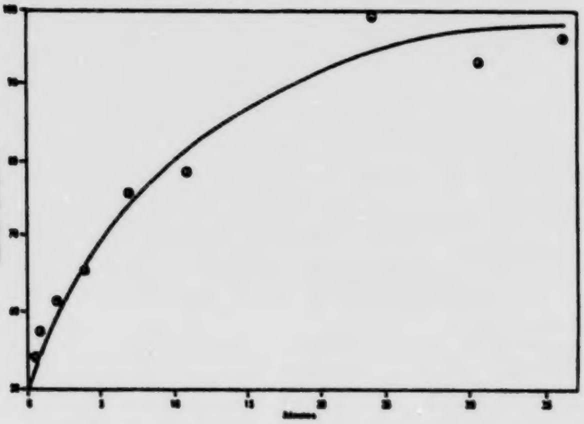
5 A.



Particle size effect on the infrared absorbance spectrum of 100 μg of standard quartz: (*a*) 8.00 μm; (*b*) 6.50 μm; (*c*) 5.20 μm; (*d*) 3.75 μm; (*e*) 2.35 μm; and (*f*) 1.70 μm. These values are the equivalent spherical diameters below which 50%, by mass, of the sized fractions occur

From: Foster and Walker (1984)

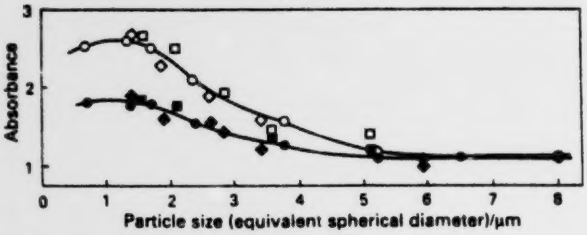
5B.



Plot of peak height of 1085-cm⁻¹ band of quartz versus grinding time. (From Painter *et al.*, 1978a.)

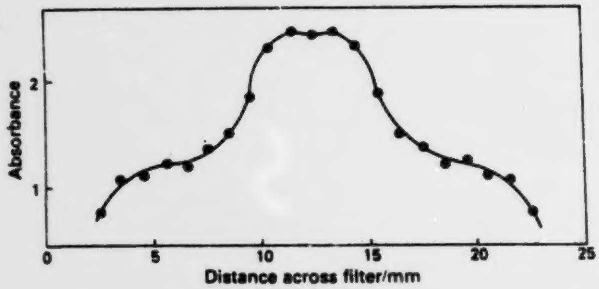
From: Jenkins and Walker (1978)

5C.



Relationship between particle size and the quartz infrared absorbance ratios at 780/790 and 799 790 for this work (●, ○) and previous work by Dodgson and Whittaker[58] (■, □) and Gade and Luft[53] (◆, ◇)

5D.



Mass distribution of sampled quartz dust across a 25 mm diameter DM800 membrane filter

b. QUARTZ BY INFRARED

Three infrared procedures have been developed by the U.S. Bureau of Mines for analysis of quartz in respirable coal dust (Freedman 1978):

1. Mine Safety Appliances (MSA, 1974):

Respirable dust is collected on 35mm MSA FWS B membrane filters and analysed directly in a Perkin-Elmer Model 267 grating infrared spectrophotometer with a beam expander. The quartz peak is corrected for kaolinite. The method is rapid but with such poor sensitivity and precision that it has been abandoned for upgraded environmental standards (Freedman, 1978, p. 319).

2. Method 19 (U.S.B.M., 1969) Standard A7 (MESA, 1971):

Up to 10 membrane filters are composited and ashed at 800°C to yield 0.40 ± 0.20mg sample. The ash is pelletized with 36 mg of potassium bromide at high pressure and under vacuum. Readings on sample and reference KBr are taken with a double beam infrared spectrophotometer and correlated to a calibration curve based on 8-50mg quartz standard pellets.

This method claims ± 10.2% precision and in 1978 was routine for some 22000 regulation samples per year.

3. Standard Method 46 (U.S.B.M., 1974):

Field samples are ultrasonically isolated and redeposited on a 10mm diameter Gelman DM450 membrane filter.

Scans from 850 to 700 cm -1 are taken for samples and calibration standards using a conventional infrared unit. Pure quartz standards prepared at concentrations of 25-150mg/cm2 in isopropanol give a calibration curve. The reported precision is ± 5% but accuracy is unknown (Friedman 1978).

In 1981, the Pittsburgh Health Technology Center revised the MESA Standard A7 procedure for respirable coal mine dust to incorporate low temperature plasma ashing (Kacsmar and Tomb, 1984). A single collection filter is ashed by LTA and resultant residue is deposited on vinyl metricel filters. A single filter loading of ≥ 0.5mg can be quantified for quartz by infrared spectroscopy.

Kacsmar and Tomb (1984) have reported on two factors that influence the above procedure: the nature of quartz standards, and the influence of an amorphous layer on quartz particles due to LTA treatment, including amorphous silica. Different standards yielded results within 10% for up to 250 ug loadings.

Foster and Walker (1984) conducted a comprehensive study of silica determination in respirable dust using a computer interfaced differential scanning I.R. spectrophotometer. Their survey included evaluation of filter types, particle size effects, heterogeneous filter loadings, sample re-radiation effects, and mineral interferences. Their selected procedures used the following:

1. Perkin-Elmer 580A differential scanning I.R. spectrophotometer interfaced to a DEC PDP 11/05 minicomputer with dual disc systems and hard copy output.
2. Gelman Sciences DM800 acrylonitrile-PVC copolymer filters: 25mm diameter 0.8um pore size.
3. Membrane filters were preconditioned by heating at 50°C.
4. Collection on-site was via a BCIRA cyclone elutriator (Cassella Ltd. type A7650/1).
5. Samples were loaded in batch sequence in rigid film holders and scanned from 400-1500 in-1.
6. Sample filters with eccentric dust depositions were scanned several times by rotation through 60-90 increments. and the spectra were averaged.
7. Standard spectra for interfering minerals were subtracted.
8. Quartz doublet peaks at 78 and 799 in-1 were monitored and compared to calibration graphs based on Sikron F-600 commercial quartz standard.

Their use of computer processing of the spectral data permitted data averaging from multiple scans as well as spectral refinement. They quoted an analytical range of 5ug to 4100ug and standard deviations of 0.8% to 4.4% for a single filter, dependant on loadings.

The study by Foster and Walker (1984) has identified other factors which influence the quality of I.R. analysis as follows:

1. Scanning the same filter across different diameters produces differing spectra, regardless of filter type. They recommend scanning a filter at known orientation before sampling dust to allow optimum filter cancellation.
2. The mass of filters is sensitive to atmospheric humidity. PVC based filters and cellulose acetate filters recorded weight changes of 0.04 to 0.07mg and 0.25mg respectively over a 36.5 to 50% humidity range.
3. Cellulose acetate filters are unsuitable for I.R. because of a strong continuium in the quartz range.
4. Materials of equivalent chemical composition but different particle size have significantly different I.R. spectra.

   The absorbance of respirable size quartz is dependent on particle size which differs across a filter loading because of the aerodynamics of collection. Figure 5d shows the change in absorbance across a 25mm membrane filter.
5. Sample re-radiation effects are severe in the presence of iron oxides, attentuating up to 75% of the silica signal to produce "an almost unrecognizable spectrum".

   This factor will be important for respirable dust from Eastern Canadian coals because of their high pyrite content. If samples are concentrated by LTA, the creation of Fe2O3 will degrade the analysis.

From the foregoing, it must be concluded that I.R. analysis of the mineralogy of respirable coal dust is not a routine procedure at present. Development of procedures suitable for the sample suite of this study was considered beyond the current scope.

NEUTRON ACTIVATION ANALYSIS

a. INTRODUCTION

Instrumental neutron activation analysis (INAA) is the standard technique for modern multielement geochemical analysis (Haskin, 1980). A sample to be analysed is sealed in a polyethylene capsule and exposed to neutron radiation in the core of a nuclear reactor. A small fraction of each isotope in the sample will react with neutrons to produce radioisotopes. These radioisotopes decay with characteristic energies and half-lives which are measured via a gamma ray spectrometer. The identities of the radioisotopes allow an identification of the parent elements.

The INNA technique is quantitative and applicable to the simultaneous analysis of up to 74 of the 83 stable elements. The detection limits are dependent on the nature of sample, specifically the background matrix. For a benign hydrocarbon matrix such as coal, detection limits are up to 10 times lower than for silicate rock (Table 14). Precision is generally ± 5% at the 97.5% confidence level (Atomic Energy of Canada Ltd. brochure).

The minimum requirement for instrumental neutron activation is a nuclear reactor with a flux of 1×10^{12} n/ cm^2/second and a Ge(Li) detector with a multichannel analyser. To be able to study

Table 14a

COAL ANALYSIS

Elements and Detection Limits by Neutron Activation

	Detection Limits		Detection Limits
Al	0.01%	Mg	0.05%
As	0.2 ppm	Mo	0.4 ppm
Au	1 ppb	Mn	0.1 ppm
B	0.2 ppm	Na	20 ppm
Ba	20 ppm	Nd	0.5 ppm
Br	0.5 ppm	Ni	50 ppm
Ca	0.1%	Rb	5 ppm
Ce	0.5 ppm	Sb	0.1 ppm
Cl	10 ppm	Sc	0.05 ppm
Co	0.1 ppm	Se	0.5 ppm
Cr	0.2 ppm	Sm	0.05 ppm
Cs	0.1 ppm	Sr	50 ppm
Dy	0.5 ppm	Ta	0.1 ppm
Eu	0.05 ppm	Ti	30 ppm
Fe	0.01%	Th	0.1 ppm
Gd	0.5 ppm	Tb	0.05 ppm
Ga	20 ppm	U	0.01 ppm
Hf	0.1 ppm	V	0.5 ppm
I	1 ppm	W	1 ppm
K	0.1%	Yb	0.01 ppm
La	0.1 ppm	Zn	3 ppm
Lu	0.01 ppm		

From: Neutron Activation Services Brochure (1984)

Table 14b

Accuracy for NBS SRM1632a and detection limits of INAA (μg/g except where noted)

Element	NBS Certified	Germani et al., 1980	This Work	Detection Limit	Element	NBS Certified	Germani et al., 1980	This Work	Detection Limit
Na	840	850	780	1	Sb	(0.6)	0.6	0.6	0.1
Cl		790	800	20	I		1.8	1.8	0.2
K		4200	3800	10	Cs	(2.4)	2.6	2.5	0.05
Sc	(6.3)[1]	6.8	6.0	0.01	Ba		122	115	20
Cr	34.4	34	32	0.5	La		18	16	0.1
Mn	23	32	26	0.1	Ce	(30)	32	24	1
Fe(%)	1.11	1.16	0.98	0.005	Sm		2.1	2.8	0.01
Co	(6.8)	6.5	6.5	0.01	Eu	(0.54)	0.55	0.52	0.01
Ni	19.4	26	18	10	Tb		0.36	0.29	0.1
Zn	28	31	29	5	Dy		2.2	2.1	0.05
Ga	(8.5)	8	8.9	0.2	Yb		0.98	1.0	0.05
As	9.3	10.2	9.8	0.1	Lu		0.18	0.19	0.05
Se	2.6	2.6	2.7	0.1	Hf	(1.6)	1.5	1.5	0.1
Br		41	41	0.1	Ta		0.40	0.35	0.01
Rb	(31)	29	31	5	W		0.6	0.8	0.2
Sr		84	81	10	Au(ng/g)			< 20	2
Mo			< 6	0.4	Th	4.5	4.8	4.1	0.1
Ag			< 0.3	0.2	U	1.28	1.2	1.1	0.1
In(ng/g)		36	33	5					

[1] Information values in parenthesis, not certified.

From: Cahill and Mills (1983)

the longer-lived isotopes, a usable neutron flux of 1×10^{12} to 1×10^{14} is necessary such as from the 5 megawatt pool-Type reactor at McMaster University. In contrast, the reactor at Dalhousie University is a SLOWPOKE model capable of only 1.0×10^{12} at 20kw (Chatt and Katz 1980).

The advantages of INAA over other multielement techniques are as follows:

1. Samples can be liquids, solids, or powders.
2. No prechemistry or postirradiation chemistry is required, therefore, reagent introduced contaminants are avoided.
3. The sensitivity to trace elements is lower than any other method, although it is function of neutron flux, irradiation time, detector resolution, sample size and matrix.

A full discussion of neutron activation techniques applicable to the geosciences can be found in Muecke (1980). In that volume, Jervis (1980) summarizes the applications to multi-element environmental studies, including airborne particulate matter.

The application of INAA to coal has been well documented. Cahill and Mills (1983) published trace element data for Australian bituminous coals including a report on the NBS SRM 1632a standard (Table 14b) for 37 elements. His work involved the TRIGA Mark II reactor at the University of Illinois which should typically deliver a neutron flux of 1.8×10^{12} at 250kw (Chatt and Katz, 1980).

Palmer and Filby (1984) analysed coal LTA from Ohio for 28 elements using a 1.0 MW reactor with a thermal neutron flux of 6 x 10^{12} n $cm^{-2}S^{-1}$. Gamma ray spectra were accumulated after various decay times from 3 minutes to 4 weeks. Their work identified trace element distributions among various size and density fractions correlated to mineralogy by SEM-EDX. This work, therefore, has correlation to our present study on respirable coal dust. No INAA study specific to coal dust was located in our literature survey.

OUR STUDY

For INAA, three commercial facilities are available in Canada as follows:

1. Atomic Energy of Canada Ltd., Radiochemical Co.
   (Isotope Products Group)
   413 March Road, Box 13500
   Kanata, Qntario K2K 1X8

2. Becquerel Laboratories Inc.
   6620 Kitimat Rd., Unit 4
   Mississauga, Ontario L5N 2B8

3. Nuclear Activation Services Ltd.
   Nuclear Research Bldg.
   1280 Main Street West
   Hamilton, Ontario L85 4K1

Only AECL and NAS have their own reactor facilities. Only NAS advertises a specific "coal analysis" package with stated detection limits (Table 14a). For purposes of this project, arrangements were, therefore, made with NAS for multielemental analysis of filters.

Samples of filter splits were preweighed and sealed in vials (supplied by NAS) at the Atlantic Coal Institute using a Mettler UM3 microbalance reading to ± 0.1µg. Vials were transmitted to the NAS reactor facility on the McMaster University campus for batch processing. Results are given in Appendix II-10a.

TRANSMISSION ELECTRON MICROSCOPY (TEM)

Harris _et al_ (1977) studied the minerals in coal down to 200 Å particle size using a scanning transmission electron microscope (STEM). Samples were prepared as optical thin sections of whole coal using ion milling and a liquid nitrogen cold stage to prevent thermal damage. By use of a microprobe of 15 Å diameter and electron diffraction , they were able to identify a variety of minerals averaging from 1 to 0.1um. Stehlow _et al_ (1978) conducted a similar TEM study concentrating on inclusions in vitrinite down to the limits of resolution (4nm).

Finkelman and Gluskoter (1981) identified the application of TEM for the following situations:

1. Mineral grains below 1um diameter.
2. If crystal structure information is desired.

They conclude, however, that TEM and STEM systems have received little routine application for coal mineral characterization because of the difficulty of extrapolating sub-micron particle data to bulk samples. Hsieh and Wert (1983) and Wert and Hsieh (1983) likewise conclude that TEM data must be supplemented by conventional SEM and XRD to make bulk conclusions.

Wert and Hsieh (1983) attempted TEM studies on coal fly ash and fine powders but experienced difficulties in sample preparation:

1. Embedding of coal particles in metal foil for ion milling failed because the coal powder floated on the evaporated metal film.

2. Ion milling of whole coal created voids in the coal and phase changes in minerals such as pyrite to pyrrhotite and clay decomposition.

From the above, we conclude that TEM would not be an appropriate technique for routine respirable dust analysis.

SEM-WDS, EMP, SSMS, etc.

Although the amended request for proposal of August 2, 1984, identified a number of other analytical techniques, our response of August 10, 1984 indicated financial and time limitations to a full evaluation. The objectives of the current work are to establish routine procedures which preclude the use of technology not readily accessible by industry in Canada.

Whereas SEM-EDX systems are now common in many Canadian industrial and consulting laboratories, wave length dispersive systems (WDS), spark source mass spectrometers (SSMS), and other non-routine instruments are rather rare on account of complexity, costs and only specialized application.

Wave-length dispersive systems of microanalysis would not be appropriate to coal dust because of resolution. WDS systems rely on light optics systems for accurate focusing on flat specimens. The limited resolution of the light microscope would not allow the visual location of specific particles. Therefore, WDS electron microprobe systems (EMP) have seen only limited use in coal minerals analysis (Finkelman and Gluskoter 1981).

Sharkey and McCartney (1981) have summarized the application of spark source mass spectrometry (SSMS) to coal mine dust. Both trace elements and organic constituents can be investigated. The method has high sensitivity and can survey a large number of trace elements with a single spectrum (see our Figure 3).

These authors report on a study of respirable dust from Pennsylvania coalfields including 56 elements. Elements that showed differences in concentration in respirable dust versus coal included Ag, Cd, Cu, Zr, Rb, Ca, Cl, P, Br likely due to the contribution of stone dust or roof rock. Respirable dust samples also showed a high contamination by saturated hydrocarbons, likely from mine machinery.

SCANNING ELECTRON MICROSCOPY AND ENERGY DISPERSIVE XRAY (SEM-EDX)

a. COAL MINERALS: SEM-EDX

Stanton and Finkelman (1979) have reviewed the characterization of both coal minerals and macerals by SEM-EDX. For the study of minerals, the SEM is ideal by virtue of magnification range, resolution, and atomic number sensitivity. Macerals, on the otherhand, are indistinct when polished pellets are observed and require optical microscopic characterization. Finkelman and Gluskoter (1981) have reviewed the problems of mineral characterization in coal by a variety of techniques. They conclude that:

> "The scanning electron microscope equipped with an energy dispersive xray detector is a nearly ideal tool for the purpose of studying the minerals in coal".

Nevertheless, the limitations to SEM-EDX were also cited:

1. Manual SEM-EDX systems analyse individual particles rather than bulk samples. Characterizing a single coal is very time intensive.
2. Computer controlled SEM-EDX Image Analysis systems can analyse thousands of particles but ignore the mineral-maceral textures.
3. Low atomic number elements are overlooked by conventional EDX (F, O, C, B, N, Be, Li).
4. Mineral identifications are based on morphology and major element chemistry. Isomers cannot be distinguished.

b. SEM-EDX-AIA

The inherent difficulty in quantitative analysis of such complex material as the mineral-maceral associations of bituminous coal led several investigators to automated image analysis (AIA) and computer controlled techniques. Lee, Huggins, and Huffman (1978) used a beam control system and software based EDX to conduct SEM characterization of minerals in washed coals. Mineral identification was based on characteristic spectra of 10 major elements supplemented by Mossbäuer analyses. A refinement of their procedures was presented by Huggins, Kosmack, Huffman and Lee (1980). Their procedure used epoxy pellets of coal observed by an inverted backscatter electron image (BEI). Two second xray counts are accumulated for 11 major elements: Na, Mg, Al, Si, P, S, Cl, K, Ca, Ti, and Fe. A computer routine classifies the particles into 29 categories (Table 15). This combined SEM-EDX and image analysis system has recently been offered commercially by Tracor Northern Inc. (Hamburg 1984), (Table 16). A flow sheet for the procedure was given by Lee and Kelley (1980) (Figure 6).

Moza, Austin, and Johnston (1979), Moza et al (1980), and Moza and Austin (1983) described a similar computer controlled technique of coal mineral analysis. Sieved coal particles are impregnated in a barium spiked resin to give a high contrast under backscatter imaging. Xray counts are collected for several points

Table 15

F. E. Huggins, D. A. Kosmack, G. P. Huffman, et al.

Coal Mineralogy Categories Utilized in SEM-AIA Program

Name	Formula/Comment	Typical EDS Percentages
Quartz	SiO_2	Si > 85
Kaolinite	$Al_2Si_2O_5(OH)_4$	Al:Si $\sim$ 45:55
Illite*	$K(Al,Fe)_4(Si,Al)_8O_{20}(OH)_4$	Al:Si:K $\sim$ 30:55:10
Chlorite*	$(Fe,Mg,Al)_6(Si,Al)_4O_{10}(OH)_8$	Mg:Al:Si:Fe $\sim$ 5:20:35:40
Montmorillonite*	$(Ca,Na)(Al,Mg)_4(Si,Al)_8O_{20}(OH)_4$	(Na,Ca):Al:Si $\sim$ 5:25:65
Gypsum	$CaSO_4 . 2H_2O$	S:Ca $\sim$ 45:55
Sulfur	S	S > 70
Jarosite	$(Na,K)Fe_3(SO_4)_2(OH)_6$	(Na,K):S:Fe $\sim$ 10:40:45
Jar/Pyr	Mixture of jarosite and pyrite	
Pyrite	FeS_2	S:Fe $\sim$ 65:30
Halite	NaCl	Na:Cl $\sim$ 15:80
Sylvite	KCl	Cl:K $\sim$ 45:55
Siderite**	$FeCO_3$	Fe > 80.
Calcite	$CaCO_3$	Ca > 80
Dolomite	$CaMg(CO_3)_2$	Mg:Ca $\sim$ 25:70
Ankerite	$Ca(Fe,Mg)(CO_3)_2$	Mg:Ca:Fe $\sim$ 10:55:30
Rutile	TiO_2	Ti > 70
Fe Sulfate	$FeSO_4 . xH_2O$ (x = 1, 4 or 7)	S:Fe $\sim$ 50:45
Apatite	$Ca_5(PO_4)_3(OH)$	P:Ca $\sim$ 25:70
Sil/Sul	Silicate-sulfur mixture	Si,S both > 20
Sil/Pyr	Silicate-pyrite mixture	Si,S > 20, Fe > 0.4S
--	Two spare categories for special use only	
Mix Sil	Mixed or unknown silicates	
Mix Sul	Mixed or unknown sulfates or sulfides	
Mix Chlor	Mixed or unknown chlorides	
Mix Carb	Mixed carbonates	
Non-int	Highest peak in EDS not one monitored by computer	
Unknown	EDS incompatible with any of above groups	

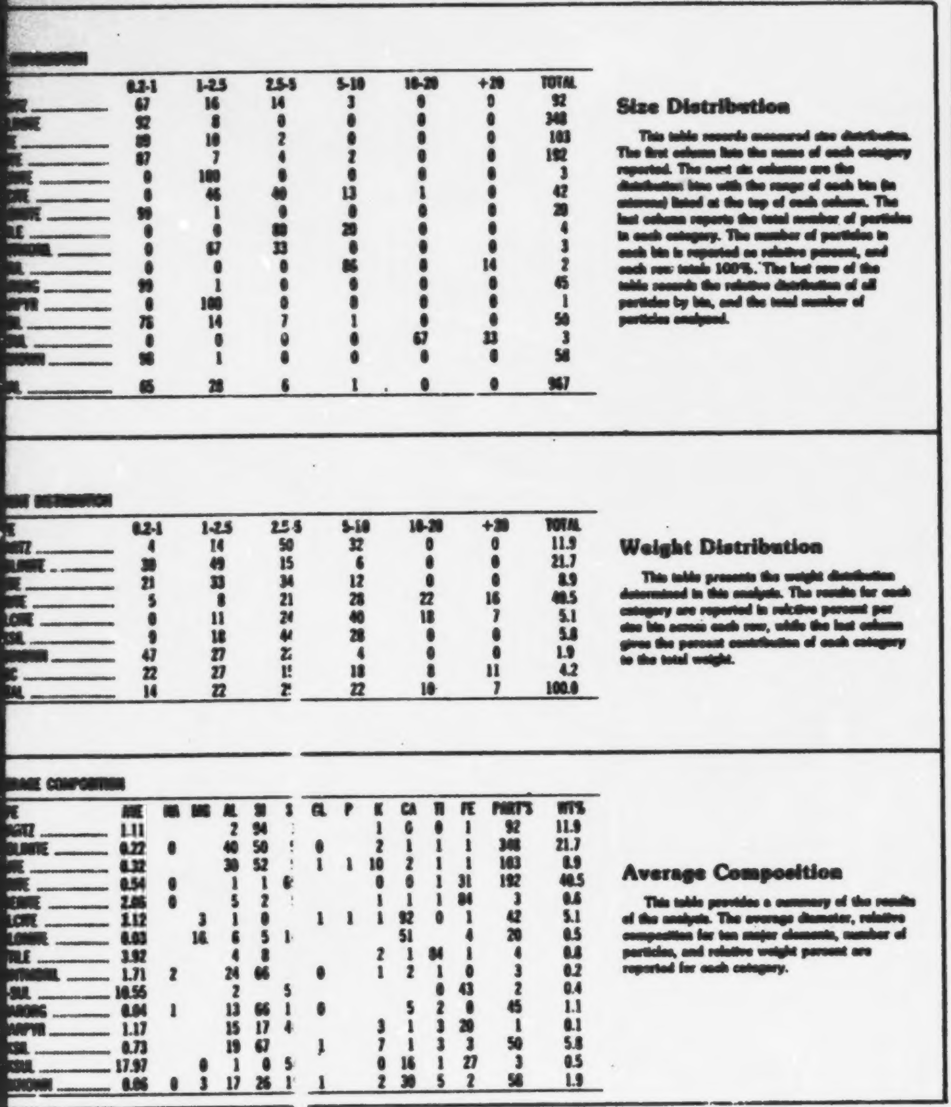
EDS -- energy dispersive spectrum.

*Formula somewhat simplified.

**Can be iron oxides or oxyhydroxides in oxidized coals. Distinguished by Mossbauer spectroscopy.

Table 16

Coal Minerals Analysis (CMA)



[illegible]	0.2-1	1-2.5	2.5-5	5-10	10-20	+20	TOTAL
[illegible]	67	16	14	3	0	0	92
[illegible]	92	8	0	0	0	0	348
[illegible]	89	10	2	0	0	0	103
[illegible]	87	7	4	2	0	0	192
[illegible]	0	100	0	0	0	0	3
[illegible]	0	46	40	13	1	0	42
[illegible]	99	1	0	0	0	0	20
[illegible]	0	0	80	20	0	0	4
[illegible]	0	67	33	0	0	0	3
[illegible]	0	0	0	86	0	14	2
[illegible]	99	1	0	0	0	0	45
[illegible]	0	100	0	0	0	0	1
[illegible]	78	14	7	1	0	0	50
[illegible]	0	0	0	0	67	33	3
[illegible]	98	1	0	0	0	0	56
[illegible]	65	28	6	1	0	0	967

Size Distribution

This table records measured size distribution. The first column lists the name of each category reported. The next six columns are the distribution bins with the range of each bin (in microns) listed at the top of each column. The last column reports the total number of particles in each category. The number of particles in each bin is reported as relative percent, and each row totals 100%. The last row of the table records the relative distribution of all particles by bin, and the total number of particles analyzed.

[illegible]	0.2-1	1-2.5	2.5-5	5-10	10-20	+20	TOTAL
[illegible]	4	14	50	32	0	0	11.9
[illegible]	30	49	15	6	0	0	21.7
[illegible]	21	33	34	12	0	0	8.9
[illegible]	5	8	21	28	22	16	40.5
[illegible]	0	11	24	40	18	7	5.1
[illegible]	9	18	44	28	0	0	5.8
[illegible]	47	27	[illegible]	4	0	0	1.9
[illegible]	22	27	[illegible]	18	8	11	4.2
[illegible]	14	22	[illegible]	22	10	7	100.0

Weight Distribution

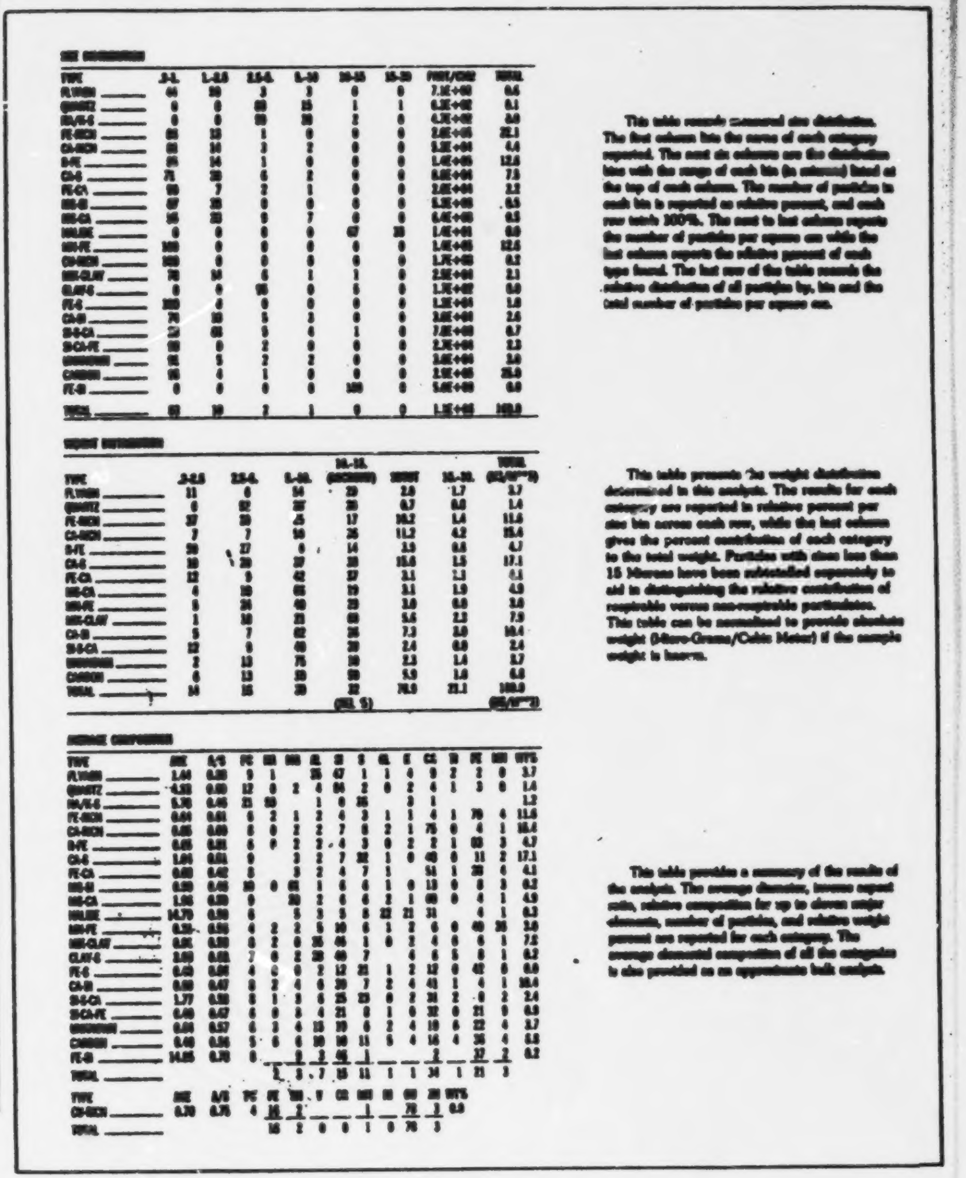
This table presents the weight distribution determined in this analysis. The results for each category are reported in relative percent per size bin across each row, while the last column gives the percent contribution of each category to the total weight.

[illegible]	AVE	NA	MG	AL	SI	S	CL	P	K	CA	TI	FE	PART'S	WT%
[illegible]	1.11			2	94	[illegible]			1	6	0	1	92	11.9
[illegible]	0.22	0		40	50	[illegible]	0		2	1	1	1	348	21.7
[illegible]	0.32			30	52	[illegible]	1	1	10	2	1	1	103	8.9
[illegible]	0.54	0		1	1	6			0	0	1	31	192	40.5
[illegible]	2.05	0		5	2	[illegible]			1	1	1	84	3	0.6
[illegible]	3.12		3	1	0		1	1	1	92	0	1	42	5.1
[illegible]	0.03		16	6	5	1				51		4	20	0.5
[illegible]	3.92			4	8				2	1	84	1	4	0.8
[illegible]	1.71	2		24	66		0		1	2	1	0	3	0.2
[illegible]	10.55			2		5					0	43	2	0.4
[illegible]	0.04	1		13	66	1	0			5	2	0	45	1.1
[illegible]	1.17			15	17	4			3	1	3	20	1	0.1
[illegible]	0.73			19	67		1		7	1	3	3	50	5.8
[illegible]	17.97		0	1	0	5			0	16	1	27	3	0.5
[illegible]	0.06	0	3	17	26	1	1		2	30	5	2	56	1.9

Average Composition

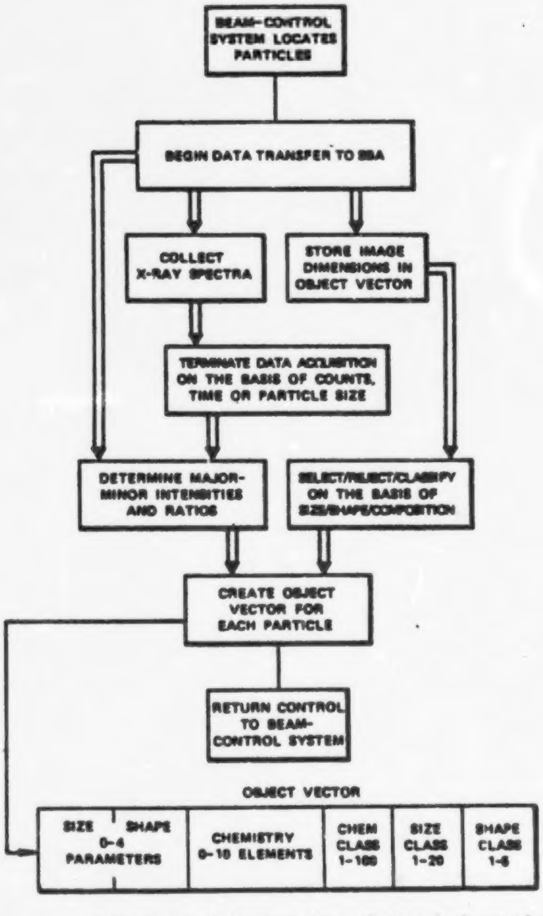
This table provides a summary of the results of the analysis. The average diameter, relative composition for ten major elements, number of particles, and relative weight percent are reported for each category.

Air Particulate Analysis (APA)



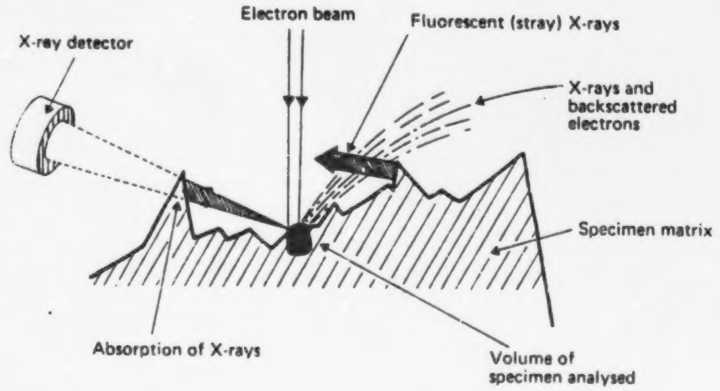
From: Tracor Northern Brochure (1982)

R. J. Lee and J. F. Kelly (1980)


BEAM-CONTROL SYSTEM LOCATES PARTICLES
BEGIN DATA TRANSFER TO SBA
COLLECT X-RAY SPECTRA
STORE IMAGE DIMENSIONS IN OBJECT VECTOR
TERMINATE DATA ACQUISITION ON THE BASIS OF COUNTS, TIME OR PARTICLE SIZE
DETERMINE MAJOR-MINOR INTENSITIES AND RATIOS
SELECT/REJECT/CLASSIFY ON THE BASIS OF SIZE/SHAPE/COMPOSITION
CREATE OBJECT VECTOR FOR EACH PARTICLE
RETURN CONTROL TO BEAM-CONTROL SYSTEM
OBJECT VECTOR
SIZE
SHAPE
0-4 PARAMETERS
CHEMISTRY 0-10 ELEMENTS
CHEM CLASS 1-100
SIZE CLASS 1-20
SHAPE CLASS 1-6

Schematic illustration of the information flow in the AIA at U.S. Steel Research.

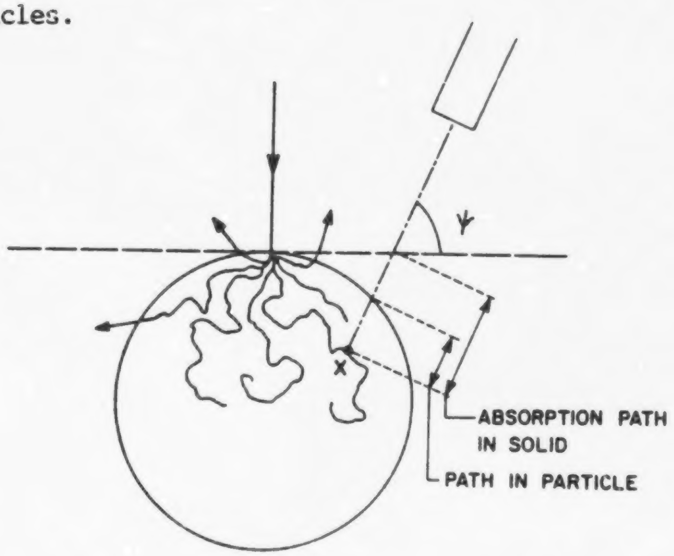
Figure 6


Electron beam
Fluorescent (stray) X-rays
X-ray detector
X-rays and backscattered electrons
Specimen matrix
Absorption of X-rays
Volume of specimen analysed

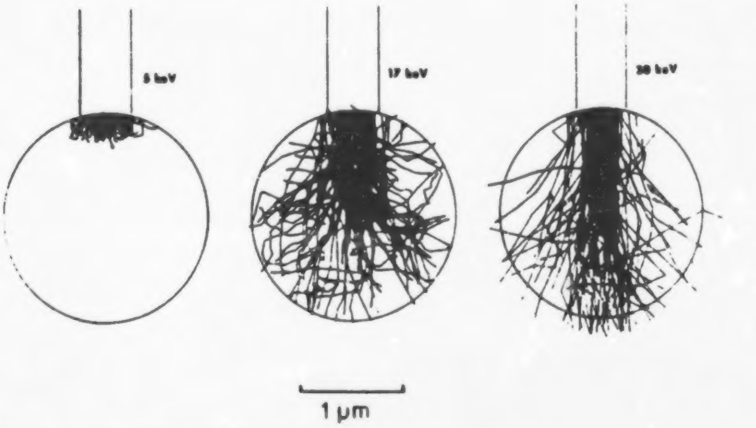
The effect of surface roughness on X-ray collection.

Figure 7.
Geochemtry effects on
EDX ray analysis of particles.

From: Chandler (1977)
p. 392.


ψ
X
ABSORPTION PATH IN SOLID
PATH IN PARTICLE

Schematic diagram comparing the absorption path in a particle with equivalent path in a bulk target.


5 keV
17 keV
30 keV
1 μm

Monte Carlo electron trajectory calculations of the interaction volume as a function of beam energy in spherical particles of aluminum

From: Goldstein et al (1981) p. 339.

on a particle and ZAF corrections applied to five elements (Al, Si, S, Ca, Fe) with others (N, O, H, Na, etc) treated as C by difference. The technique is restricted to the size range 8-75um and is designed to give "average" particulate chemistry for a size class, rather than mineralogical information. Moza, Strickler, and Austin (1980) refined the technique to include particulate classes. Their particle size range (>8um) is not appropriate for respirable dust samples.

c. COAL DUST: SEM-EDX

Application of scanning electron microscopy to respirable coal mine dust dates from the work of DeNee (1971) and Thaulow and White (1971). Thaulow and White (1971) reported on a method of coal mine dust dispersion in naphthalene-camphor in preparation for SEM study. Their procedure required some 20mg of powder sample. DeNee (1971) used bulk dust and filter loadings obtained from four different collector devices and subjected them to various sample preparations. DeNee (1971) identified the following minerals and elements by SEM-EDX using a working magnification of x3000 and a particulate range of 0.1um to 10um:

Minerals

pyrite (FeS_2)
magnetite (Fe_3O_4)
quartz (SiO_2)
limestone $(CaCO_3)$
dolomite $(MgCa(CO_3)_2)$
gypsum $(CaSO_4.2H_2O)$
anhydrite $(CaSO_4)$

Elements

Na, Mg, Al, Si, P, S, Cl, K, Ca, Mn, Fe

DeNee *et al* (1973) published the first SEM demonstration of respirable dust *in situ* in the human lung using autopsy tissue from three stages of coal workers' pneumoconiosis. Mukherjee and Dixon (1983) likewise used SEM-EDX to study coal miner lung cases.

DeNee's (1971) work identified some of the technical problems and solutions to SEM-EDX analysis of respirable coal dust including the following:

1. Uncoated dust can be viewed by collection on gold coated glass.
2. Nuclepore filters are the most appropriate for "as-captured" viewing.
3. The mean particle size of 1 - 1.5um is best viewed at a magnification of x3000.

His work did not attempt to quantify chemical or morphological results. Lebiedzik *et al* (1973) and Troutman *et al* (1974) were the first to apply computer processing of SEM images to respirable coal dust. Their technique was the forerunner to the work of Moza and Austin (1983) although the later authors limited their work to 10-100um coal particles. Lebiedzik *et al* (1973) used a Germanium substrate to calculate particle thickness from the intensity of the GeK_{α} line using A1203 sputtered film, carbon spheres, and glass fibres as standards.

Operating at 25kv they characterized 0.1 to 10um particles by the xray intensities of 5 elements: Al, Si, S, Ca, and Fe. This, they claimed to be sufficient to distinguish between the major particle types in bituminous coal mine dust. Table 17 shows their particulate classification and morphological information. The following mineral types are listed:

TYPE	ELEMENT
coal	--
Al2O3	Al
Quartz	Si
Kaolinite	Al, Si
Calcite	Ca
Illite/feldspar	Al, Si, K
Gypsum	S, Ca
Iron oxide	Fe
Pyrite	S, Fe
Mica	Al, Si, Ca, Fe
Rock fragments	Other combinations

Johnston et al (1981) used a similar approach to atmospheric particulate matter, although not specifically coal mine dust. Teflon membrane filters from dichotomous air samplers with loadings of 50-100ug/cm2 were analysed by both bulk XRF and computer assisted SEM-EDX. For the SEM work, samples were carbon coated and analysed at 30kv for 500 to 1000 particles. Their particle classifications are shown in Table 17b. Their work showed good agreement of XRF chemistry to normalized particle elemental weight percents especially for major elements Al, Si, K, Ca. Halogens Cl and Br gave lower results by SEM-EDX than by XRF suggesting a loss of elements at high vacuum (Johnson et al 1981, p. 474). Minor elements Mn, Cu, and Zn showed variable results because they were non-homogeneously distributed on a particle by particle basis.

The works of Lebiedzik et al (1973) and Johnston et al (1981) have shown the potential for particle-by-particle analysis of respirable dust. The present state-of-the art is, however, exemplified by the commercial system of Tracor Northern Inc. based on the work of the U.S. Steel Research Laboratories (Lee et al 1978, Huggins et al 1980, Kelly et al 1980, Lee and Kelley 1980, Hamburg 1984). Based on a new generation of xray microanalysers and computer controlled image analysis, their programs for coal mineral and air particulate analysis (Table 16) provide a greater array of information than any predecessors. Combining these two programmes should allow for a customized particulate analysis specific to coal mine dust.

The refinement of particle-by-particle analysis is still in progress. Kelly et al (1980) identified conditions necessary for reliable results from automated particulate analysis:

1. Particle overlap should be minimal (<5%).
2. Background should be smooth and featureless. Only membrane filters (eg. Nuclepore) are appropriate, although Teflon filters show promise.
3. Better contrast of particle to background is required for automated than manual particulate analysis.
4. Backscattered electron mode gives usable contrast down to 0.5um particle size.
5. Filter samples must show homogeneous loadings.

Table 17. From: Lebiedzik _et al_ (1973)

Table 1. Particle Type Identification Used in CESEMI Analysis of Coal Mine Dust

	IDENTIFICATION	CHANNEL 1 SEC. ELEC.	CHANNEL 2 ALUMINUM	CHANNEL 3 SILICON	CHANNEL 4 SULFUR	CHANNEL 5 CA	CHANNEL 6 IRON
0	COAL	X					
1	AL2O3	X	X				
2	QUARTZ	X		X			
3	KAOLINITE	X	X	X			
4	ROCK FRAGMENT	X			X		
5	ROCK FRAGMENT	X	X		X		
6	ROCK FRAGMENT	X		X	X		
7	ROCK FRAGMENT	X	X	X	X		
8	CALCITE	X				X	
9	ROCK FRAGMENT	X	X			X	
10	ROCK FRAGMENT	X		X		X	
11	ILLITE/FELDSPAR	X	X	X		X	
12	GYPSUM	X			X	X	
13	ROCK FRAGMENT	X	X		X	X	
14	ROCK FRAGMENT	X		X	X	X	
15	ROCK FRAGMENT	X	X	X	X	X	
16	IRON OXIDE	X					X
17	ROCK FRAGMENT	X	X				X
18	ROCK FRAGMENT	X		X			X
19	ROCK FRAGMENT	X	X	X			X
20	PYRITE	X			X		X
21	ROCK FRAGMENT	X	X		X		X
22	ROCK FRAGMENT	X		X	X		X
23	ROCK FRAGMENT	X	X	X	X		X
24	ROCK FRAGMENT	X				X	X
25	ROCK FRAGMENT	X	X			X	X
26	ROCK FRAGMENT	X		X		X	X
27	MICA	X	X	X		X	X
28	ROCK FRAGMENT	X			X	X	X
29	ROCK FRAGMENT	X	X		X	X	X
30	ROCK FRAGMENT	X		X	X	X	X
31	ROCK FRAGMENT	X	X	X	X	X	X
32	TOTAL OF ALL ROCK FRAGMENTS						

Table 2. Summary Table for Coal Mine Dust Sample for CESEMI Analysis

PARTICLE TYPE	DENSITY GRAMS PER (CM**3)	NUMBER OF PARTICLES	STATISTICAL NUMBER OF PARTICLES	PERCENT OF TOTAL NUMBER OF PARTICLES	AREA OF PARTICLES SQUARE MICRONS	PERCENT OF TOTAL AREA OF PARTICLES	AVERAGE SIZE AREA SQUARE MICRONS	AVERAGE EQUIVALENT CIRCULAR DIAMETER MICRONS	WEIGHT OF AVERAGE PARTICLES 10**-12GM	WEIGHT % OF AVERAGE PARTICLES	WEIGHT OF ACTUAL PARTICLES 10**-12GM	WEIGHT % OF ACTUAL PARTICLES
0	1.30	709	709	81.00	6097.3	82.23	8.60	3.31	17535.4	61.6	48384.2	73.7
1	4.00	7	7	0.81	38.3	0.52	5.48	2.64	269.9	1.0	291.3	0.4
2	2.65	29	29	3.37	431.2	5.82	14.87	4.35	3319.1	11.7	4832.3	7.4
3	2.65	5	5	0.58	96.4	1.30	19.28	4.95	853.6	3.0	1406.6	2.1
4	3.00	17	17	1.97	124.2	1.68	7.31	3.05	750.0	2.7	1442.2	2.2
6	3.00	3	3	0.35	3.6	0.05	1.19	1.23	6.7	0.0	10.0	0.0
8	2.72								1054.4	3.7	1629.0	2.5
12	2.30	1	1	0.12	11.6	0.16	11.56	3.84	68.0	0.2	68.0	0.1
14	3.20	18	18	2.09	247.8	3.34	13.77	4.19	3596.6	12.7	6454.4	9.8
18	3.00	1	1	0.12	19.8	0.27	19.76	5.02	198.5	0.7	198.5	0.3
20	5.00	1	1	0.12	0.8	0.01	0.82	1.02	2.9	0.0	2.9	0.0
24	3.00	7	7	0.81	53.1	0.72	7.59	3.11	330.2	1.2	426.2	0.6
25	3.00	1	1	0.12	31.1	0.42	31.11	6.29	391.6	1.4	391.6	0.6
30	3.00	29	29	3.37	231.8	3.13	7.99	3.19	1670.8	5.9	2449.1	3.6

Table 17b

Atmospheric particulates by computerized SEM-EDX:

Chemistry Definition File in Specific Gravities

Class	Category Name	Specific Gravity Assigned
1	Low Counts	1.100
2	Marine Aerosol	2.165
3	Sec. Type	1.600
4	CA+S	3.000
5	Wood Comb.	1.900
6	Wood Refuse	3.000
7	Comb. Cars	2.500
8	Comb. Oil	3.200
9	Combustion Lg.	1.500
10	Combustion	2.400
11	Industrial	4.600
12	Dolomite like	2.870
13	Calcite like	2.700
14	Construction	2.450
15	Asbestos/Talc type	2.500
16	Illite like	2.600
17	Kaolinite like	2.650
18	Feldspar Pl like	2.500
19	Mica B like	2.800
20	Mica M like	2.850
21	AL Rich	3.960
22	SI Only	2.330
23	Lithophilic	2.750
24	Misc. Salts	1.550
25	Misc. Class	2.500

Particle Elemental Stoichometry by Chemistry Definition File Class in Table I

Element	Molecular Form Used in Computations
Na	Na_2O - all classes
Mg	MgO - all classes
Al	Al_2O_3 - all classes
Si	SiO_2 - all classes
P	PO_4 - all classes
S	SO_4 - classes 1-9; S - classes 10-25
Cl	Cl - all classes
Cd	CdO - classes 1-5; Cd - classes 6-11;
	CdO - classes 12-25
K	K_2O - all classes
Sn	SnO - classes 1-5; Sn - classes 6-11;
	SnO - classes 12-25
Ca	CaO - classes 1-4, 6-11; Ca metal - class 5;
	$CaCo_3$ - classes 12-25
Ti	TiO_2 - all classes
V	V_2O5 - all classes
Cr	Cr_2O_3 - classes 1-10, 12-25; Cr - class 11
Mn	MnO_2 - classes 1-10, 12-25; Mn - class 11
Fe	FeO - classes 1-10, 12-25; Fe - class 11
Co	CoO - classes 1-10, 12-25; Co - class 11
Ni	NiO - classes 1-10, 12-25; Ni - class 11
Cu	CuO - classes 1-10, 12-25; Cu - class 11
Zn	ZnO - classes 1-10, 12-25; Zn - class 11
Pb	PbO - classes 1-3, 6-7, 9-10, 12-25;
	Pb - classes 8, 11; PbO_2 - classes 4,5
Br	Br - all classes

From: Johnson et al (1981)

6. At least 100 particles per category are required for quantitative results.
7. No single particle should account for more than 2% of total sample area.

If the contrast (ie. signal/noise ratio) is insufficient, the results will be distorted because of missed particles or textured grains being recorded as multiple particles. The time required for a reliable analysis is sample dependent and can range from 30 minutes to several hours of computer controlled SEM-EDX time.

CANDIDATE ANALYTICAL METHOD: SEM-EDX

INTRODUCTION:

We previously discussed the published literature on SEM-EDX applied to coal minerals and respirable coal dust. The following section covers our in-house facilities and the procedures applied to this study. Compared to the previous authors, we had the advantage of considerable improvements in instrumentation, especially with regard to data processing. The in-house facilities at the Atlantic Coal Institute include:

1. JEOL T300 Scanning Electron Microscope with variable 1-30kv accelerating voltage, 15x to 200,000x magnification and guaranteed resolution of 6nm (60 Å). Photomicrographs are obtained from a 2000 line ultrahigh resolution CRT using a Polaroid 545 land camera (4"x5") and PN55 film.
   Samples are loaded on aluminium holders as stubs (brass, aluminium, carbon), pellets etc. and mounted on a eucentric stage. Imaging is by secondary electrons (S.E.I.) or a high sensitivity annular type backscattered electron detector (B.E.I.).

2. Tracor Northern 5500 System Xray Microanalyser including:
   - a. Microtrace, 30mm2 area, 154ev resolution, horizontal Si(Li) detector.
   - b. DEC LSI 11/23 minicomputer with 256K byte CPU, dual 8" disk drives, microprocessor based 4096 channel data memory with 16 million count capacity.
   - c. High resolution colour monitor with ASCII keyboard control console.
   - d. Model 8510A C.Itoh Electronics Inc. 120 cps dot matrix printer
   - e. Quantitative microanalysis software including standardless ZAF corrected analysis, Bence Albee geological analysis, Micro Q digital filtering and ZAF analysis.

3. Polaron E5100 series II cool sputter S.E.M. coating system with:
    a. annular gold target
    b. E5000-374 carbon rod evaporator
    c. E5000-376 carbon fibre evaporator
4. Structure Probe Inc. SPI Plasma Prep II low temperature asher with an Edwards 5 two stage rotary pump.

This equipment allows for several procedures not routinely found in SEM laboratories, including the following applicable to our study:

1. Sputter coating of samples at low temperature via a thermoelectrically cooled stage (4°C).
2. Low temperature ashing of coal matrix (<150°C) via radiofrequency generation of oxygen plasma.
3. Full quantitative energy dispersive xray microanalysis at state-of-the art resolution and data processing.

PARENT MATERIAL: R.O.M. COAL

Procedures

The technique adopted for R.O.M. coal samples was based on the experience of Lee et al. (1978), Finkelman and Stanton (1978), Stanton and Finkelman (1979), Huggins et al. (1980), and Raymond and Hagan (1982). Representative -20 mesh coal samples were mounted as 25mm diameter acrylic lucite pellets using a Metaserv moulding unit. Pellets underwent grinding on three Metaserv polisher-grinders using 240, 400, and 600 grit adhesives followed by polishing stages with cloth/3.0um alumina powder and silk/0.05um alumina powder in distilled water. This procedure follows ASTM D2797-72 and results in a fine polished surface suitable for optical or electron microscopic observation.

To prevent beam charging, pellet mounted samples were coated with an atomic layer of carbon using a fiber filament in a Polaron SEM coating system. Electroconductive connections were made between pellet and aluminum holder using Jeol Dotite XC-12 paint. The pellet and holder were introduced into the sample chamber of the JEOL T300 scanning electron microscope. After a 3 minute pump down (rotary and diffusion pumps), the instrument was set to 20kv with a working distance of 10-20mm. After initial focus imaging and stigmation corrections in the secondary electron image mode, the backscattered electron detector was activated and maximum contrast established between macerals and minerals. Photographs were taken with Polaroid PN55 film at 90 second exposures on the high resolution 2000 line CRT.

Energy dispersive xray microanalysis was performed with the Si(Li) detector in. Spectra were generally collected for 50 seconds and plotted on line by dot matrix printer/plotter.

The EDX system is capable of detecting all elements from sodium (Z=11) to uranium (Z=92) in the Periodic Table. Elements below Na are not detected because of the presence of a beryllium window protecting the Si(Li) detector. Therefore, the system can differentiate most minerals except for the following:

1. Isomers such as pyrite (FeS2) and marcasite (FeS2).
2. Minerals differing only in light elements H, Li, Be, B, C, N, O, and F.
3. Hydrated species (CaSO4 versus CaSO4.2H2O)

Mineral identifications were based on the major element spectral characteristics identified by Welton (1984) and Huggins _et al._ (1980) (repeated by Hamburg, 1984). Certain assumptions had to be made based on this published experience. Kaolinite and illite were assumed to be the major clays, rather than their spectral isomers: halloysite and muscovite. Likewise "pyrite" was preferred over "marcasite" unless morphological evidence existed for sectored or radial crystals. "Siderite" was preferred over "magnetite or hematite" even though the EDX couldn't differentiate oxides from carbonates. These assumptions are, however, supported by extensive petrographic evidence of Sprunk and O'Donnel (1942) regarding mineral abundances in coal.

TABLE 18

MINERALS IN RUN-OF-MINE COAL FROM SYDNEY COALFIELD

	MINE/SEAM:	Lingan/Harbour	Novaco/Harbour	Novaco/Harbour	Prince/Hub	Brogan/Lloyd
	SAMPLE:	001	E 010	W 011	003	009
TEKTOSILICATES:	Quartz	A1	D1	DC	DC	D2
PHYLLOSILICATES:	Kaolinite	DC,D1	*	*	DC,E1,A1	D2
	Illite	DC,D2	DC	DC	D2	D1,E1
	Biotite	*	*	*	D2	D1
	Montmorillonites	*	*	*	*	*
	Chlorite	*	*	*	D2	*
	Fe-Cu-silicate	*	*	*	D2	*
NESOSILICATES:	Zircon	*	*	DC	*	*
HALIDES:	Halite	*	*	*	*	x
CARBONATES:	Calcite	*	*	*	*	*
	Ankerite	DC	*	*	D2	*
	Siderite	D1	*	*	*	D2
SULPHIDES:	Pyrite	A1,E1,DC	E1	E1,DC	E1,A1,DC	DC,D1,E1,A2,A1
	Marcasite	*	*	*	*	*
	Pyrrhotite	*	*	DC	*	in pyrite
	Pentlandite	*	in pyrite	*	*	*
SULPHATES:	Gypsum	*	*	*	*	*
PHOSPHATES:	Apatite	*	*	*	*	*
	Monazite	*	*	*	*	*
OXIDES:	Magnetite	E2	*	*	*	*
	Corundum	x	*	*	*	*
	Rutile	*	*	A1	*	D1

LEGEND:	Detrital-	D1 in coal	D2 from run-of-mine rock	DC in Carbominerite
	Authigenic-	A1 primary	A2 secondary, overgrowths	in inclusion
	Epigenetic-	E1 fillings	E2 weathering	x contaminant or artifact.

MINERALS IN SYDNEY COAL

Table 18 is a compilation of all minerals characterized to date in R.O.M. samples from the Sydney Basin. The following additional petrogenetic observations were made.

Illite is the dominant clay in carbominerite grains of the Harbour Seam and Lloyd Cove. Kaolinite occurs as fusinite cell fillings at Lingan (Harbour Seam) and Prince-Hub. Quartz is common as inclusions in Harbour Seam coal but occurs only as run-of-mine rock contributions for the Hub and Lloyd Cove Seams. Traces of syngenetic ankerite are identified for the Lingan-Harbour, but detrital (R.O.M.?) grains occur in the Hub.

Pyrite habit is variable for all samples including massive infills, lone octahedra, framboids, and crystallite colonies with particulates to submicron diameters. No marcasite was recognized.

A single detrital zircon contemporaneous with carbominerite bedding and a lone rutile rod was identified in a Novaco-Harbour West sample, but many other such accessory minerals would be revealed with more analytical work.

The Prince-Hub sample appeared to have the most diverse roof rock contribution to R.O.M. as evidenced by detrital biotite and chlorite as well as the major minerals of quartz and illite.

A full documentation of minerals in the Sydney coalfield was considered beyond the scope of the present study. Therefore, we have elected in photomicrographs and EDX spectra, to illustrate general findings for Eastern Canadian coals as apply to respirable dust. The reader is refered to the Appendix II, section 1. These photos and EDX spectra illustrate several features of note:

1. Respirable dust size particles of pyrite and clay photos 2, 3, 4, 6, 10).
2. Flocculation of clays and generation of amorphous material during low temperature ashing (photos 3, 4).
3. Presence of rare minerals and trace element inclusions (photos 12, 18).
4. Angular and elongate morphology of coal macerals (photos 19,1).
5. Variable pyrite morphology (photos 4, 6, 5, 6, 10-16) and cementation (photos 16, 10).

Of special note for particulate analysis is photo 14 with corresponding replicate EDX traces 18-27. These spot analyses on various crystal faces of pyrite octahedra illustrate the problem of geometric effects on xray microanalysis. The resultant EDX spectra show variable ratios of Fe/S which could lead to mineral misidentification.

TRACE ELEMENTS IN SYDNEY COAL

Finkelman (1982a, 1982b) and Finkelman (1978) used the SEM analysis of accessory minerals to define the trace element sites in coal. His work of 1978 concluded that titanium was largely bound organically. A complete table of his suggested modes of occurence is reproduced in Table 2.

Our work has revealed the following minor and trace element associations based on individual mineral analyses.

Element	**Association**
Na	montmorillonite, halite
Mn	ankerite, siderite, chlorite
Mg	ankerite, chlorite, tremolite
Ti	illite, organic, biotite rutile, chlorite, pyrite
P	apatite, monazite
Cu	pyrite, siderite, illite Fe silicate (?), biotite, organic
Cl	organic, halite, illite ankerite, chlorite
Ni	pyrite
Ce	monazite
Zr	zircon

In addition, we have analysed coal macerals within carbominerite or maceral structures distinct from the pellet matrix. The EDX spectra of macerals are generally dominated by the Si and S peaks over a large background. Secondary peaks of Al and Fe are ubiquitous but other peaks present are dependent on the coal seam. The Sydney Coalfield, for example, is characterized by generally high Ti and Cl or K.

PARENT MATERIAL: STONE DUST

We previously presented the typical suite of analyses conducted on stone dust as used at the CBDC mines (Table 8). The limited analysis for oxides suggest primarily calcite (CaO: 52%; CO2: 43%) with traces of dolomite or ankerite (MgO: 2%) and quartz (SiO2: 3%).

For the present study, we received one sample of stone dust of unknown origin as used at the CBDC mines at present. How similar this material is to the analyses of Table 8 is unknown. Nevertheless, we expect a similarity in chemistry and particle size (-200 mesh).

Photomicrographs (#1 and #2) show a rather uniform particle size but a variable chemistry. Eight EDX spectra (#'s 1 - 8) identify a complex mineralogical suite of at least 9 minerals. Complete quantitative analysis is considered beyond the scope of the present study. Nevertheless, the variety of minerals suggests a similar diverse contribution to respirable dust. A simple identification of the stone dust contribution on any one filter loading is not likely easy for coal mine dust if similar carbonates and sulphates are present in the coals or roof rock.

STANDARD REFERENCE MATERIAL

INTRODUCTION

Filby et al (1985) have discussed the problem of standardization and quality control in methods requiring submilligram sample sizes. They evaluated various geochemical standard reference materials including the NBS coal (SRM1632) and coal fly ash (SRM1633, 1633a) for replicate INAA analysis using various sample weights. The SRM1632 coal was the most heterogeneous standard analysed with relative standard deviations>20% for Sb, Ni, Ba, U, Zr, and Tb.

The problem of standards for microanalysis is even more severe when considering electron microscopic techniques for which the analytical beam diameter is in the order of 1 to 2 microns. Jarosewich and MacIntyre (1983) mentioned the problem of specimen degradation under a microprobe of such minerals as calcite, aragonite, and dolomite. Jarosewich et al (1980) described 26 minerals prepared for use as microprobe standards but cautioned that even after the most careful preparation, grains of accessory minerals or matrix remain. They addressed the problem by assigning a "homogeneity index" from multiple spot analyses on ten grains.

The 1982-83 CANMET Review made reference to working groups established to address practical reference standards for calibration of airborne contaminants. The status of such groups or the application of their work to respirable coal dust was not known to us at the time writing.

The National Bureau of Standards (U.S. Department of Commerce) issued a calibration standard entitled "Urban Particulate Matter" (SRM 1648) based on baghouse collection in St. Louis, Missouri. We acquired this material for the present study but found it unsuitable for several reasons: limited analytical information, heterogeneity and composition unrelated to our coal problem.

A more suitable NBS standard is SRM 1878 "Respirable alpha quartz" (95.5%) designed for quantitative xray powder diffraction analysis. This material is the correct particle size (0.33 to 5um), but the suppliers warn that it is not intended for use as a particle size reference material because of agglomeration. More applicable to our sample suite is the "Quartz on Filter Media" standard package (SRM 2679a) based on 2 to 202ug loadings of SRM 1878 quartz in 370mg of SRM 97a flint clay. The certificate identifies several limitations:

1. Non-uniform quartz distribution
2. Clay content not certified
3. Quantitative results for quartz must use the entire filter.

OUR STUDY

As discussed previously, the mineralogical and chemical analysis of coal dust is a multivariant problem. Reference standards should, therefore, address the range of material encountered. To this end, we utilized three sources:

1. Astimex Scientific Limited (Toronto, Ont.)
   MINM25-53 Mineral mount.
2. Chemplex Industries Inc. (Eastchester, N.Y.)
   XRD Mineralogy Set.
3. National Bureau of Standards
   SRM 2685 coal; SRM 1878 quartz; SRM 2679a quartz on filter; SRM 1648 urban particulates.

SRM 2685 COAL

We included the NBS coal standard in our sample suite submitted for neutron activation analysis (Appendix II-10). The 32 elements listed by the Certificate of Analysis were not certified, but since they were based on INAA calibration against NBS 1632a and 1635, they should be useable. No mineralogical information was available for this standard, therefore, no SEM-EDX evaluations were made.

SRM 1878 QUARTZ

Photomicrograph #1 (Appendix II 2a) shows the particle size distribution suitable for respirable dust studies. Agglomeration is evident when dry dispersed on Nuclepore. The EDX spectrum shows a monomineralic character (SiO_2).

SRM 2679a (BI). QUARTZ/CLAY ON FILTER

This standard, suitable for quartz analysis by XRD is not appropriate for microprobe particulate analysis. The gum tragacanth solution matrix interfers with morphological observations of particles by SEM. Furthermore, the gum and filter give background peaks of Cl, Ca, Fe. We also identified a titanium-rich contaminant (EDX #4, Appendix II 2a).

SRM 1648 URBAN PARTICULATES

This standard is suitable for bulk analysis (>100mg) of atmospheric particulate matter. The Certificate of Analysis indicates a comprehensive suite of trace elements analysed by INAA but no particulate information. Although it could act as an analytical INAA standard, the lack of mineralogical information and poor chemical correlation to our expected coal dust chemistry precludes reliance on this standard for our project.

CHEMPLEX XRD MINERALOGY SET

The Chemplex Industries Inc. mineralogical powders were obtained specifically to allow the preparation of standard mixes based on known minerals. A total of 50 minerals are included in the set as -250 mesh powders prepared by isodynamic separation techniques. These standards are advertised to be "pure" and suitable for a variety of instruments including microprobe work.

Our investigations have revealed that the Chemplex standards are heterogenous mineral mixes not suitable for particulate analysis. Photomicrographs (Appendix II 2c) show a variety of particulate textures and contrasts indicating several mineral species. The microcline feldspar standard revealed barite, iron oxide, a rare earth mineral, and plagioclase feldspar (?). The calcite, biotite, and siderite likewise yielded a host of different EDX spectra when particle-by-particle analysis was applied (EDX spectra 1-21, Appendix II 2d).

ASTIMEX SCIENTIFIC LTD. MINM25-53

To identify the wide variety of possible mineral species, we relied on published spectra (Welton 1974) as well as a set of spectra generated on our in-house system using the 53 mineral microprobe standard from Astimex. The Astimex standards are mounted as polished chips in a single (53 mineral) 25mm epoxy resin pellet. The minerals were selected for within-grain homogeneity and have been quantitatively analysed at the Universities of Toronto and Alberta. Appendix II 2e gives sample spectra for 18 minerals more commonly associated with coals. Several minerals (e.g. illite, ankerite, pyrite) prevalent in Sydney Basin coals are not included. Their recognition was based on published experience since standards are not stable (Jarosewich and MacIntyre 1983; Faye and Sutarno 1976) or not available.

When the reported chemistry for the Astimex standards is compared to our EDX spectra, the absence of minor or trace element peaks gives a guide to our detection limits. Table 19 lists the maximum and minimum elemental concentrations missed or detected from the 18 mineral spectra shown in the appendix. We conclude that elements present in concentrations >0.6% are generally no problem unless there are extensive peak overlaps. For overlapping peaks and rare elements, concentrations as high as 8.7% have been missed.

TABLE 19. EDX DETECTION LIMITS*
(this study)

Element	EDX #	Lowest Contentration Detected (wt%)	Highest Concentration Not Detected (wt%)
Al		10.34	0.10
Ba		58.84	0.01
Ca		29.38	0.40
Ce		30.8	---
Cr		0.67	0.14
F		48.67	0.02
Fe		72.19	0.66
K		8.23	0.34
La		12.9	---
Mg		13.18	0.08
Mn		---	0.03
Na		3.23	---
Nd		---	8.7**
Ni		---	0.19
P		12.1	---
Pb		86.6	---
Pr		---	4.2**
S		13.74	---
Si		14.04	---
Sn		---	0.45
Sr		---	0.07
Tn		3.8	---
Ti		1.06	0.04
V		---	0.38
Zn		65.68	---

*Based on 18 mineral standards in polished epoxy mount, 50 second counts.

**Overlapping peaks.

The foregoing problems with available mineral powder standards led us to abandon the search for reference material suitable for addition or dilution methods or generation of calibration curves. The main efforts to analyse respirable dust were, therefore, directed towards particle-by-particle recognition or qualitative "fingerprinting" the mineralogical or chemical character of the filter loadings. The numerical data generated from such point counts or rasters are independent of the need for reference standards except for qualitative comparisons.

PARTICULATE PREPARATIONS

Filter Substrates

As previously cited, DeNee (1971) and Kelly et al. (1980) identified the substrate requirements for successful SEM analysis of coal particulates to include a featureless background, high particle contrast and homogeneous particulate distribution with no overlap. Nuclepore filters were recognized as the most appropriate for "as captured" dust analysis. Foster and Walker (1984) did a similar survey for the best filter material for instrumental I.R. analysis of coal dust and identified the need for:

1. High collection efficiency.
2. Moisture resistance.
3. Low electrostatic charging.
4. Flat analytical background (for I.R. and XRD).

For SEM-EDX analysis, we must add the requirement for low atomic number composition ($Z<11$) to avoid interfering EDX peaks.

Gelman and Meltzer (1979) discussed appropriate filters for coal dust collection in personal samplers. They recommended 5um vinyl chloride or acrylonitrile copolymer filters for collection of 1um to 9um particles because of the non-hygroscopic character and low ash content. For redeposition of coal dust, they suggested vinyl chloride with a 0.45um porosity.

Filter types made available to us included cellulose acetate (Millipore), polyvinyl chloride (PVC), glass fibre (MSA), silver membrane and Nuclepore of various diameters and pore sizes (Table 4). In order to minimize sample preparation and allow "as captured" analysis, we conducted a survey of these filter types to define the best collection medium.

The Nuclepore filters (Appendix II 3a, photo #5) exhibited the lowest surface relief and one of the more benign Xray backgrounds (EDX traces #2). Note, however, that Murphy (1982) reported abundant trace elements. Nuclepore is a 10um thick polycarbonate film in which fission fragment tracks have been etched out to leave highly uniform cylindrical pores. It can withstand temperatures up to 140°C and is soluble in chloroform, ethylene dichloride and strong bases but resists alcohols, dilute acids, and hydrocarbons (Frank et al., 1970). For electron microscopy of particles, it has long been recognized as a superior substrate (Johari and DeNee 1972, Chatfield and Dillon 1978) especially for automated quantitative work.

Chatfield and Dillon (1978) have indicated that redeposition of particles onto Nuclepore by filtration is very difficult and usually leads to uneven loadings. Johari and DeNee (1972) have indicated charging problems at the pores which can be overcome by precoating with carbon. We, in our study, have identified handling problems with Nuclepore because of electrostatic charging and the filter flexibility. Under the SEM beam, particles tend to move on Nuclepore (also reported by Murphy 1982).

Millipore type filters of cellulose acetate represented most of the filters received from CANMET personnel for our study (Table 4). As shown by photos (Appendix II 3a, 19-20), the sponge-like matrix structure of Millipore traps and masks individual particulates and is, therefore, unsuitable for "as captured" viewing by SEM. Similar reservations were voiced by Chatfield and Dillon (1978). The EDX spectrum for Millipore cellulose is relatively featureless but shows a large background continuum (EDX #5, Appendix 3b). Murphy (1982) reported no elemental interference, but we detected Si and Cu, possibly from our instrument.

Glass fibre filters were the least suitable for SEM-EDX study because of the fibrous network (photos 1-4) and the strong interfering peaks of Si, Al, Ca, and Mg (EDX #1). Likewise, EDX interference was shown for silver membrane (Ag and trace Na, Si, Cl) and PVC (Cl) as illustrated by EDX spectra #3, 9, and 10.

The suitability of filters for field collection was studied by CANMET personnel on site at CBDC mines (Dan Kennedy, personal communication). Pressure drop characteristics showed that Nuclepore filters could be used down to 0.8um pore diameter. Handling problems are overcome by using a backing filter as a rigid support.

Chatfield and Dillon (1978) do not recommend Nuclepore filters for field sampling. They suggest the use of Millipore followed by low temperature ashing and redeposition onto Nuclepore.

Their work, however, concentrated on asbestos fibres. For our work on coal dust, we must consider the LTA mineral transformations that would occur even for short ashing times. Alternate methods of redeposition were mentioned in DeNee (1971).

In consultation with the Scientific Authority, it was decided that field sampling must be dictated by logistics which favoured Millipore rather than optimum SEM conditions which favoured Nuclepore. The bulk of the work concentrated on "as captured" analysis using the submitted filters as received. The objectives to develop a rapid routine methodology required this compromise on sample quality.

FILTER MOUNTS

Two types of sample holders have been used for the direct mounts of respirable dust filters. For larger filter sections, a 2" or 4" aluminium disc is covered with Lomacoll Double Coated Mounting Film (Lohmann GMBH and Co., Neuwied, W.Germany). The filter is directly adhered and subsequently sputter coated. This Lomacoll film is carbon based and masks the underlying aluminium from the electron beam.

For smaller filter sections, we use a 20mm diameter carbon planchette (Marivac catalogue #95117-0) or a 10mm carbon stub (Marivac catalogue #AS113-0). The filter is adhered with "sticky tabs" (Marivac catalogue #AS102) adhesive film, and subsequently sputter coated.

To ensure electroconductive connections, we paint filter edges and connecting bridges across the stub and holder interface using JEOL Dotite XC12 carbon paint. All such work is done in a Gallenkamp glove box to prevent extraneous aerosol dust contamination.

The dust loadings on filters from the personal samplers varied considerably across the filter diameters. Representative samples were, therefore, obtained by cutting a pie shaped wedge. The Nuclepore filters proved especially fragile. A scalpel blade, rolled across the filter on a glass substrate provided a clean break.

The resultant SEM images obtained for various loadings and operating conditions are illustrated in Appendix II 3c.

PARTICULATE MOUNTS:

DeNee (1971) mentioned sampling methods for coal dust involving removal from collection filters and aspiration onto gold coated glass slides or other substrates. Particulate isolation from web like filters was by ultrasonic cleaning in an alcohol solution. Substrates included cellophane tape, cellulose nitrate film, and a beryllium stub.

Thaulow and White (1971) described a procedure to disperse coal dust onto glass slides using a eutectic mixture of camphor and naphthalene (60:40) which sublimes under vacuum without leaving a residue.

Other investigators of particle handling techniques for SEM-EDX investigation include Johari and DeNee (1971), Chatfield and Dillon (1978), and Murphy (1982). These authors propose such a variety of substrates, adhesives, tapes, etc. and procedures of filtration and dispersion that a full evaluation could not be made in the time constraints of this project. We, therefore, elected a limited investigation as described in the following sections.

SLURRY MOUNTS:

A sample of Carbogel (coal water slurry) was obtained from the Cape Breton Development Corporation. An aliquot of this mixture was dispersed in a quantity of distilled water which had been previously filtered through a 0.22um Millipore filter. The simplest method of mounting such a sample is to evaporate a droplet of the above mixture on the surface of a polished SEM stub. For this purpose, we prepared standard aluminium, brass and stainless steel stubs by polishing on a metallurgical Metaserv grinder/polisher using diamond pastes.

Even after ultrasonic cleaning, the polished metal surfaces proved poor for particulate analysis. Residual polishing powders were observed as well as remnant scratches which interfered with particle images (Appendix II 3d, photo #23). Furthermore, the metal substrates generated significant interfering Xray peaks (Fe, Al, etc) precluding qualitative spectral recognition.

The simple evaporation of solutions also suffers from uneven particulate loading due to the Marangoni effect. As the liquid evaporates the larger particles settle while smaller particles are transported by the evaporating edge.

SPUTTER COATING:

DeNee (1971) reported that respirable coal dust particles do not need to be coated if collected on gold coated glass slides or cellophane tape, but membrane filters require electroconductive coatings. We have found excessive beam charging for all substrates unless gold or carbon coats are applied. Even with coatings, the irregular particulate surfaces showed considerable image charging artifacts in the SEI mode.

Initially, we depended on gold coating techniques because a more continuous electroconductive film could be achieved with rough surfaces. By the end of the project we had perfected carbon coating techniques which are preferable for xray microanalysis.

Gold coating was carried out in a Polaron E5100 Series II Cool Sputter Coater using argon to flush the chamber. The specimen chamber is pumped down to 0.02mbar, and the sample stage is cooled to 5°C. Coating is by an annular gold anode at a voltage of 2.5kv and a current of 18mA. The thickness of Au coats were monitored visually with white paper inserts and controled by varying the sputtering times from 60 seconds to 2 minutes. The thickness can be calculated as 135 Å to 270 Å based on:

$th = 7.5\ I \times t$ where: th = thickness in angstroms
I = mA current
t = time in minutes
target to specimen distance: 50mm

The length of sputtering times was based on the filter loading topography. Lightly loaded Nuclepore filters exhibited no charging after 60 second coatings, but Millipore filters still showed some charging at 2 minutes.

Carbon coating proved to be more difficult in practice, especially for the rough topography of Millipore filters. The carbon fibres supplied with the Polaron E5000-376 Carbon Evaporation Attachment did not perform as well as subsequent fibres purchased from Marivac (catalogue #VE141-0). The previous material exhibited severe outgasing.

A vacuum of 0.02 mbar is established, and the current, set at 8.5mA, is pulsed for 2 second intervals. Several filaments are required for an adequate coating which is monitored by a paper insert. We did attempt to use a carbon rod attachment but found the heat generated to be excessive which caused damage to the sample filters.

Our sputter coating system proved to be well adapted to the respirable dust filters because of two unique features: annular gold target and Peltier cooling stage, which prevent thermal damage. Echlin (1978) has evaluated coating techniques for SEM-EDX and especially the Polaron II cool sputter coater. He writes:

> "The introduction of the low-temperature sputter coater provides the most convenient, reliable and reproducible means of coating specimens..."
>
> "The electron bombardment of the specimens is significantly reduced by replacing the disc shaped target of the diode coater with an annular target.
> ...As a further precaution the specimen holder is cooled by a small Peltier effect cooling module. Using this improved equipment we have been able to coat crystalline hydrocarbon waxes with melting points as low as 32°C and thermolabile sensitive plastic films, neither of which had been previously coated by any of the methods available."

Echlin and Kaye (1979) have furthermore reported that by lowering the specimen temperature, the gold coatings are of smaller grain size and more continuous.

LOW TEMPERATURE ASH

Chatfield and Dillon (1978) have refered to a procedure of dust collection on Millipore filters followed by plasma ashing and redeposition onto Nuclepore. We felt it appropriate to evaluate plasma ashing as a possible preparative step for our respirable dust filters.

Plasma ashing or "low temperature ashing" (LTA) requires an oxygen gas plasma to rapidly oxidize the organic sample matrix without high temperature combustion. This allows for the isolation of inorganic constituents from a masking organic matrix without severely altering the components. We have previously pointed out, however, that for minerals in coal, several alterations do occur, including clay dehydration, pyrite oxidation, and the generation of amorphous artifacts.

For tests on filters, we used a Structure Probe Inc. Plasma Prep II (West Chester P.A.). Loaded filters are placed in a pyrex ashing chamber which is evaculated to 1 Torr. Oxygen is leaked in at 5 psig, and an RF power of 85 watts is applied for a period of approximately 15 hours.

Our first attempt involved one loaded Nuclepore filter ashed onto a gold coated glass slide. The resultant particulate distribution was inappropriate for SEM-EDX analysis because of flocculates and particle overlap (Appendix II, 4a photo 11).

Our next test involved a composite of 3 heavily loaded filters from the same occupational group. We wished to produce a bulk sample similar to our coal LTA for comparative SEM-EDX. The composite sample underwent LTA for 15 hours on an ultraclean pyrex petri dish. The resultant ash was dispersed in prefiltered acetone, sonicated, and filtered onto a 0.1um pore size Nuclepore filter. After gold coating, the ash underwent SEM-EDX investigation as illustrated in Appendix II, section 4a, b.

The mineral suite identified in the dust LTA includes residual coal, pyrite, kaolinite, illite, calcite, and a fibrous mineral possibly chlorite or serpentine. In addition, we identified grains of a copper-rich mineral with variable Cu:S ratios. A tentative identification is chalcocite or an oxidized product antlerite, but neither have been previously seen in the parent coals or stone dust. We suggest that this material is extraneous.

The low temperature ashing was not carried to completion, therefore, residual coal was present; nevertheless, pyrite showed partial oxidation with loss of sulphur. Pyrite framboid morphology was preserved indicating that at least some framboids are airborne intact and do not disintegrate on oxidation.

PARTICULATE ANALYSIS

SEM-EDX PARAMETERS

There are several selectable SEM-EDX instrument parameters which can be varied to suit the sample type and analytical objectives. Many of these are interrelated and will affect both SEM images and the EDX analysis.

Maggiore and Rubin (1973) discussed the optimum SEM-EDX instrument conditions applied to respirable dust samples. They used a field emission gun and a scanning transmission mode to view asbestos fibres down to 500 Å diameter. For our study, we had the advantage of improvements in electronics and computerization that allowed better results with conventional SEM-EDX and a tungsten filament source. Table 20 compiles the instrument parameters used by previous investigators for coal and respirable dust, especially coal dust. Our system differed in better resolution of both the SEM image and the Xray detection. Furthermore, we conducted all data processing on-line. The improved efficiency in electronics allowed for higher xray count rates with deadtimes up to 65% and durations of only a few seconds to produce distinctive spectra.

TABLE 20

SEM-EDX INSTRUMENT PARAMETERS FOR PARTICULATE SAMPLES

PARAMETER	DE NEE (1971)	MAGGIORE and RUBIN (1973)	LEBIEDZIK *et al* (1973)	HUGGINS *et al* (1980) and (1982)	MOZA et al (1979); MOZA AND AUSTIN (1983)	BIRK and O'NEIL (1985) this study:
SAMPLE						
Type:	coal mine dust	respirable dust	coal mine dust	coal -60 mesh .2-50um	coal particles >8um	coal mine dust
Substrate:	Nuclepore or glass + Au	TEM grid	camphor/ naphthalene on glass	epoxy pellet	Ba spiked resin pellet	Nuclepore, Millipore
Coating:	± C	C	Au or Al	C	C	C, Au,
Holder:	---	carbon	---	aluminium	---	aluminium
SEM						
Source:		field emission	---	---	---	tungsten
Max magnification:	---	x200,000	---	---	---	x200,000
Usual mag:	x3000	---	---	x600	---	x2000, x3500, x10,00
Mode:	SEI,BEI	TV	TV(?)	inverted BEI	inverted BEI	SEI,BEI
Control:	manual	manual	beam control	LeMont beam	beam control	manual
Max resolution:	---	25nm	10nm	---	---	6nm
Working distance:	---	---	---	---	---	20mm
Kv	---	18	25,40	20	20	20, 15
EDX						
Mode:	multi-channel	multi-channel	5 channels	11 channels	5 channels	multichannel spectrum
Detector type:	Si(Li)	Si(Li)	Si(Li)	Si(Li)	Si(Li)	Si(Li)
Resolution:	---	170ev	---	---	---	154ev
Collimator:	---	Delrin + C	---	---	---	aluminium + C
Electronics:	---	pile-up rejection circuits	CESEMI system	---	CESEMI system	100 MHz a/d conversion
Rate:	---	to 20,000 cps	---	---	---	15,000 - 20,000
Sample distance:	---	20mm	---	---	---	20mm
Collection:	---	20,140s	100ms	20s	2s	50,10s
COMPUTER						
Data capture:	---	DEC PDP 11/20	---	TN2000 system (PDP11)	PDP 11/20	TN5500 system (DEC LSI 11/23)
Processing:	---	offline	IBM 360 offline	online	online	online
Language:	---	---	Fortran IV	---	Fortran	Flextran
Storage:	---	magnetic tape	---	magnetic tape	---	dual disk
Software	---	---	---	ZAF	ZAF	ZAF

Most work was conducted at an SEM specimen to pole piece working distance (WD) of 20mm. This was selected to optimize high resolution imaging while preserving an appropriate xray take-off angle on 1 micron size particles.

A 20kv accelerating beam voltage was selected to give sufficient particle to background contrast in the backscattered electron mode (BEI). This voltage also matched the conditions for published mineral spectra (McCrone and Delly, 1973; Welton 1984) thereby allowing comparisons. For smaller particles (≤ 2 micron), this beam voltage results in an interaction volume for primary electrons greater than the particle mass. Since most of our investigations required only qualitative particulate recognition, this was not a severe problem. Moza et al (1979) however, considered the Xray sampling volume for coal to be as much as 5um and thereby restricted their quantitative work to >8um particles.

DeNee (1971) identified x3000 magnifications to be most appropriate for coal dust. Our system employs step-wise magnifications of which x2000 and x3500 were most appropriate to area views of respirable dust. The x3500 magnification was near the practical limit for sharp photos in the BEI mode. The secondary electron image mode (SEI) allows for much higher usable magnifications; therefore, point counts were taken at x10,000 in this mode.

In our system, the condenser lens current or beam "spot size" was adjusted to minimum settings for SEI high resolution photos but required higher currents for BEI contrast. Other SEM instrument parameters of contrast, brightness, gamma, astigmation, and focus were altered as required to optimize the images for each analysis or were assigned to the auto settings of the instrument.

For energy dispersive xray microanalysis, the Si(Li) detector position remained fixed. To produce reliable spectra on standards, we generally collected xrays for 50 seconds or more at settings giving >3000 counts per second. Count rates were altered by changing the condenser lens current, the gun bias, or the working distance. Electronic dead time varied from 25 to 65% depending on the count rates.

For qualitative characterization of a given particle, a very high count rate was prefered (15,000 to 20,000 cps) giving a large deadtime (60-65%) to allow rapid 10 second analysis or less during point counting.

DATA PROCESSING

The Tracor Northern TN5500 Xray Microanalyser used in this study is currently the most advanced commercial EDX system. Five dedicated microprocessors control data acquisition, pulse processing, multichannel scaling, xray spectra calibration, CRT display, and system diagnostics. The spectral data is displayed on a 512x256 full colour CRT allowing the operator to view 512 discrete energy channels and output to a printer/plotter. A cursor provides accurate location, KeV, and intensity of each channel while KLM markers identify element peaks from Boron to Einsteinium including their sum and escape peaks.

For our study, spectra covering the xray energy range of 0 to 10.24 KeV were displayed with vertical full scales (VFS) set to the highest peak. Although the system is capable of peak deconvolution and background subtraction, this was not routinely applicable to our particulate samples.

Software available to manipulate the resultant xray data included three levels of complexity:

1. General purpose programmes:
    a. Match/Sort: Qualitative spectral storage, match, and sorting.
    b. Ident: qualitative xray peak identification.
    c. Q-file: multifunctional programme to format the intensity data and apply statistical analysis.
2. Standardless Quantitative Analysis:

    Standardless Semi-Quant (SSQ):
    calculates element or oxide concentrations from bulk sample ZAF matrix corrections

3. Quantitative Analysis:

   a. Atomic number (Z), Absorption (A) and Fluorescence (F) corrections:
   for standards and unknowns.

   b. Bence-Albee matrix corrections for mineral oxides are applied to standards and unknowns using empirical correction coefficients.

We have elsewhere discussed the problems of quantitative particle analysis and the lack of suitable standards. The powerful software packages described above are designed either for bulk polished specimens or for infinitely thin films. The respirable dust samples complied with neither set of assumptions and, therefore, data processing using these programmes was limited.

AREA RASTER SCANS

For heterogeneous particulate samples such as our respirable coal dusts, it would be advantageous to obtain an "average" analysis by simply rastering the electron beam over a field of particles. Myklebust et al (1981) from the National Bureau of Standards (U.S.) have evaluated the significance of such "average particle compositions" based on the composite xray spectrum. They conclude that overscanning of particle mixtures should not be used, even for semi-quantitative results. Their conclusions are based on tests using particle standards, 200-400 second rasters and conventional ZAF matrix corrections:

1. Homogeneous K961 glass spheres of 1-10um size, yielded errors of -17% to +60% for 8 elements.
2. Homogeneous K961 glass shards of 1-10um size yielded errors of -11% to +80% for 8 elements.
3. A two component glass sphere mix (K309 and K227) yielded errors of -24% to +256% for 6 elements.

They concluded that even the most favorable case of homogeneous spheres yielded large errors. Heavy elements (Ti, Fe) were greatly overestimated. Even specially developed particle matrix correction procedures couldn't compensate for elements partitioned in different matrices. Spectra averaged over particle mixtures should, therefore, be used only in a qualitative sense.

At the request of the Scientific Authority, the use of area rastering for xray data acquisition was evaluated for the respirable coal dust filters. Although the technical limitations described above were recognized, it was felt that qualitative "fingerprinting" might still be appropriate.

To test how reproducible such spectra are in relation to raster size and filter area, one loaded Nuclepore filter was subjected to multiple area rastering while collecting EDX spectra.

Area scans (rasters) were made at three magnifications (x500, x1000, x2000) covering rectangular areas of correspondingly reduced fields of view. Appendix II 5a gives the spectra plus the data reduction using the quantitative analysis package SSQ of Tracor Northern. Table 21 summarizes the elemental concentrations reported by the programme, although such ZAF corrections assume flat geometry and infinitely thick samples. The various magnifications give similar results with some data scatter but no clear trends. Different areas on the filter yield only small differences in chemistry such as in Al, S, Ca which may not be statistically significant.

Xray analysis by area rastering was also applied to coal dust collected from various work stations. Table 22 summarizes the SSQ apparent elemental concentrations for 10 filter collections, with raw data presented in Appendix II 5b.

The dust LTA composite sample shows notably less chlorine content, likely due to volatile loss during ashing. A higher Ti

TABLE 21

REPLICATE EDX AREA SCANS OVER COAL DUST ON NUCLEPORE FILTER (85-15)*

NORMALIZED APPARENT ELEMENTAL CONCENTRATIONS(wt.%) **

TEST TYPE	AL	SI	S	FE	CA	K	CL	TI	CU
AREA 1 2000X	21.36	43.49	21.09	6.62	0.00	3.44	0.99	0.00	3.01
AREA 1 1000X	20.45	43.81	21.43	6.07	0.00	3.38	0.83	0.00	4.03
AREA 1 500X	20.41	43.35	21.36	6.64	0.00	3.23	1.26	0.00	3.75
AREA 2 2000X	20.35	42.00	22.22	6.38	0.01	3.39	1.75	0.00	3.90
AREA 2 1000X	20.43	42.51	22.02	6.47	0.00	2.84	2.09	0.00	3.65
AREA 2 500X	20.41	43.20	21.70	7.08	0.00	3.16	1.48	0.00	2.97
AREA 3 2000X	19.60	43.77	21.83	6.56	0.44	2.93	1.15	0.00	3.71
AREA 3 1000X	19.89	42.51	22.31	7.25	0.04	2.61	2.09	0.00	3.30
AREA 3 500X	19.89	42.52	22.31	7.25	0.04	2.61	2.09	0.00	3.30
AVERAGE	20.31	43.02	21.81	6.70	0.06	3.07	1.53	0.00	3.51
STANDARD DEVIATION	0.47	0.61	0.42	0.38	0.14	0.31	0.47	0.00	0.36

* Gold coated

**Data generated by Tracor-Northern SSQ program. Note that conditions for quantitative results were not met by our particulate samples. Area rastering is considered appropriate for qualitative comparisons only.

Table 22

EDX AREA SCANS: RESPIRABLE COAL DUST

SAMPLE TYPE	SAMPLE NUMBER	EDX NUMBER	NORMALIZED APPARENT ELEMENTAL CONCENTRATIONS(wt.%) **								
			AL	SI	S	FE	CA	K	CL	TI	CU
SHEARER(NUCLEPORE)	LTA-COMP	1	18.44	35.32	10.50	17.59	7.49	4.97	0.77	1.19	3.72
SHEARER(NUCLEPORE)	85-175	2	12.05	45.11	12.36	15.39	1.25	2.07	4.70	0.00	7.08
SHEARER(MILLIPORE)	85-53	3	13.96	37.86	6.88	13.82	15.30	3.83	3.18	0.00	5.16
GENERAL(NUCLEPORE)	85-132	4	6.12	41.83	4.17	18.78	13.58	0.00	3.52	0.00	12.00
BRUSHER(MILLIPORE)	85-39	5	9.97	71.97	3.70	6.02	0.18	0.97	1.55	0.00	5.65
BRUSHER(MILLIPORE)	85-9	6	11.84	59.93	3.18	11.52	0.09	1.93	1.49	0.00	10.02
SHOTFIRER(MILLIPORE)	85-38	7	18.54	39.36	4.89	20.59	8.03	4.18	1.31	0.22	2.87
SHOTFIRER(MILLIPORE)	85-76	8	19.41	48.88	5.97	12.73	2.78	4.40	1.39	0.00	4.44
JACKMAN(MILLIPORE)	85-98	9	17.92	49.50	4.98	13.15	3.21	4.69	1.32	0.46	4.77
CONTROL PT.(NUCLEPORE)	85-15	10	23.96	51.40	9.69	5.80	0.00	4.34	2.33	0.00	2.48
CONTROL PT.(NUCLEPORE)*	85-15	11	20.64	43.62	20.43	6.38	0.00	3.25	1.50	0.00	4.18

* gold coated; all others carbon coated

**Data generated by Tracor-Northern SSQ program. Note that conditions for quantitative results were not met by our particulate samples. Area rastering is considered appropriate for qualitative comparisons only.

value may be due to better peak differentiation with the attenuated xray continuum in the absence of organics. A gold coating in one sample gives an anomalously high sulphur value and an enhanced copper peak due to interference of gold in the peak regions of interest.

Although the number of analyses is limited, a trend related to work sites seems to be present. The shearer gets dust with a higher chlorine and sulphur content, probably because of a larger coal maceral contribution. The brusher, in contrast, receives dust with a higher silica , reduced pyrite (Fe,S) and reduced stone dust (Ca) content. This would be expected if the roof rocks contributed most of the dust.

These trends need confirmation with additional analyses and correlation to actual collection conditions. The nature of filter substrates must be considered although no correlation of elements to Millipore versus Nuclepore filters is apparent in Table 22. Other apparent trends need explanation. For example, the general mine sample 85-132 is depleted in K and Al but enhanced in Cu. Is this related to the unusual copper-rich mineralogy reported elsewhere? Is the apparent Ca enrichment in this sample due to stone dust, and if so, why is there a similar enrichment in one shearer sample? Furthermore, the qualitative nature of these area rasters must temper any conclusions. A visual comparison of EDX spectra does not support the numerical data for Cu enrichment in

85-132 but, to the contrary, suggests higher copper levels in sample 85-39. This data must, therefore, be interpreted with caution.

In addition to problems previously identified for EDX area rastering of particulates, our work has identified further limitations:

1. Specimen charging
2. Particulate loss

With heavily loaded filters, particles tend to eject from the filter surface when subject to the beam raster. This is due to excessive specimen charging, even for coated samples. The charging also creates spurious xray signals, which may contribute to analytical error.

QUANTITATIVE PARTICLE ANALYSIS

INTRODUCTION

Small (1981) has reviewed eight methods of quantitative analysis of particles by electron beam instruments and has identified the limitations. Standard electroprobe analysis using ZAF or Bence-Albee corrections requires that the samples have infinite thickness and a flat polished surface. In contrast, the uncontrolled geometry of small particles leads to several interferences compared to bulk analysis:

1. Mass Effect:

   Particle mass is finite and may be smaller than the interaction volume of the primary electron beam, leading to poor particle/background resolution.

2. Absorption Effect:

   The xray absorption path length is unknown, leading to overestimates for low energy xrays (Si,Al)

3. Fluorescence Effect:

   The secondary excitation by continuum or characteristic xrays is reduced giving underestimates for all elements.

For respirable dust size ("thin") particles (<10um), Small (1981) identified the following methods:

1. Relative Sensitivity Factor Method

   Thin particles and thin film standards are analysed with 100 keV beam energies in a TEM or analytical electron microscope and intensity ratios are calculated for element pairs from the Cliff-Lorimer equation:

$K_{AB} \cdot I_A / I_B = C_A / C_B$ where: K = constant
I = intensities
C = concentrations
A,B = two elements

From the K_{AB} determined on standards, the composition of unknown particles can be calculated.

2. Estimation of Local Mass:

This method requires that particles fit the thin-film approximation and have a low Z matrix. If the local sample mass is known for particle and standard, the concentration of an element is calculated from relative xray intensities. The local sample mass is calculated from the intensity of the continuous xray spectrum.

Both of the foregoing methods require particle standards similar in size and shape to the unknowns and, therefore, cannot use thick polished standards. Small (1981) concludes:

> "...there are several problems in using particle standards since the magnitude of the various particle effects are dependent on several parameters including particle size and shape. It is my feeling that the use of particle rather than bulk standards increases the probability of compounding errors. In addition, the availability of well characterized particle standards, especially multielement standards is limited." p.456

Small (1981) also reviewed EDX methods applicable to larger particles (>10um) including the following:

1. Normalization of ZAF corrections for bulk standards and unknown. This can apply to particles with similar energy elements or only elements of >4 keV energies.
2. ZAF corrections using particle standards. Standards and unknowns must have similar particle size and shape.

3. Normalized beam raster area. The electron beam is rastered over an area larger than the particle, and the loss in xray intensity is corrected by the area ratio (particle/raster).
4. Modified P Factor Method. Multielement bulk standards are used to analyse particles with a particle mass correction factor (P) calculated from thin film procedures.
5. Geometric Modeling of Particle Shape. A particle is assigned to a geometric shape and various particle effects are calculated.
6. Peak to Background. ZAF corrections are based on the ratio of characteristic xrays to the bremsstrahlung xrays of the same energy. Both are produced from the sample volume and will suffer similar absorption effects.

This last method is most appropriate for rough bulk samples but cannot compensate for composite spectra due to adjacent particulates (Goldstein et al, 1981).

Goldstein et al (1981) suggested that simple normalization of bulk sample ZAF corrections is appropriate for small particles (<3um) since the mass effect dominates. On the other hand, they suggested that presentation of "raw" data based on bulk sample ZAF procedures may be better. If all elements are accounted for, the discrepency of the unnormalized analysis from 100% indicates the relative errors due to particle geometry (Goldstein et al 1981, p.347).

In reviewing the foregoing options for quantitative particulate analysis, we concluded that none were appropriate to our problem of respirable dust. The following factors preclude their application:

1. Standards for coal dust are not available. Preparation of homogeneous multi-element standards with similar particle geometry is not practical.
2. Techniques employing bulk polished standards are not appropriate to <10um dust.
3. Software for complex computer calculations specific to particles ie. geometry modeling, customized ZAF corrections, etc. are not available.

For the project on hand, we, therefore, elected to limit particulate analysis to qualitative recognition or we present uncorrected bulk ZAF procedures as suggested by Goldstein _et al_ (1981). The reader is, therefore, cautioned that such results are for comparative purposes and only semi-quantitative at best.

PARTICULATE RECOGNITION

Photomicrographs of individual coal dust particles are illustrated in Appendix II 6a with corresponding EDX spectra. Minerals with distinctive chemistry are readily recognized if interference from filter substrate or gold coating is minimized. The following mineral suite is reported from a total of some 3075 particle analyses performed on our coal dust samples:

Organic Constituents: coal macerals, carbominerite

Major minerals: illite
calcite
quartz
pyrite
kaolinite

Minor minerals: chlorite
siderite or hematite
barite

Trace or ambiguous:
anhydrite, plagioclase
halite, rutile,
kyanite (?) iron silicate (?)
monazite (?) chalcocite (?)
antlerite (?), serpentine (?)

Of the 57 minerals reported by Gluskoter et al (1981) as occuring in coals, we have found 15 in the Sydney coal mine dust. In addition, we have identified four minerals not previously cited, especially the presence of copper sulphate minerals.

Of these 19 minerals, the calcite and barite are likely a stone dust contribution while kaolinite and pyrite are primarily coal derived. The relative contributions of quartz and illite from roof rock or coal mineralogy cannot be determined since these minerals are major constituents in both sources. Chlorite is likely a roof-rock contribution while the iron phase is either oxidized from pyrite (hematite) or rock derived (siderite).

Mineral EDX spectra are categorized on major peaks and ratios following Welton (1984), McCrone and Delly (1973) and others, with consideration for Xray geometric or elemental substitution effects.

Having established the ability to identify the mineralogy of respirable dust size particles, we next investigated rapid analysis of several particles in a single field of view using the

Millipore filter substrates. As previously discussed, these filters are not suitable for optimum SEM imaging. The ability to spot analyse discrete particles for short count times at moderate magnification required demonstration.

Photomicrographs 1-44 (Appendix II 7a) and EDX spectra 1-44 (Appendix II 7b) document the analysis of particles on Millipore using a x2000 magnification, carbon coating, and 10 second counts. Although the morphological distinction of particles is obscurred by the Millipore filter matrix, the chemical signatures from spot analysis are diagnostic for most mineral grains.

Coal particles are differentiated from inorganic minerals by the low count yields (small vertical full scale VFS) and the large Xray continuum background. Such spectra may, however, be confused with filter pores which likewise yield low counts. Minnis (1984) used low Xray counts of <100 to define pores versus >2000 counts for sandstone grains when 2 second EDX spot analyses were taken. We found that pores generally give even lower readings than coal and lack the distinctive Si and S peaks.

MODAL ANALYSIS BY POINT COUNT

Petrographic or "point count" analysis has been used for more than 100 years to determine the mineral distribution in a rock sample. Hutchison (1974) discussed, in detail, procedures for quantitative results based on 1000-2000 optical point counts and careful selection of the representative modal unit cell. Minnis (1984) has compared SEM-EDX automatic point counting with XRD and petrographic methods on three different sandstone samples. Results varied, partially because of identification problems with optical petrographic procedures, the higher resolution of SEM-EDX, and the poor resolution of XRD. Minnis (1984) cited 500 points as sufficient to generate an optical petrographic analysis accurate within 5% for major minerals, but her automated SEM-EDX system used a 51 x 51 grid (2601 points).

To evaluate rapid EDX identification of individual dust particles as a potential mineralogical modal analysis, we were constrained by the lack of an automatic beam control or image analysis and, therefore, manual operation was limited by operator fatigue. We therefore elected a compromise of 500 particles as representative of each filter loading. At 2000x magnification, the field of view on the CRT contains over 3000 dust particles. Manual particle selection and spot analysis was better conducted at higher magnifications (x10000). Therefore, a random selection of 500 particles by sample traverse covered only a small portion of each filter. This lack of control on the modal unit cell and the low statistical count must be considered when evaluating the manual results.

The cited problems with manual operation were, however, partially offset by several advantages. Automated SEM-EDX-AIA systems require a threshold setting in BEI mode to define particles from background. As is evident from our photomicrographs of coal dust on Millipore (Appendix II 7a), such a selection of contrast threshold is almost impossible for some of our samples. Furthermore, the manual selection of particles and beam positioning ensured that most points represented discrete particles analysed at the centroid. Unusual spectra could be stored by the operator for further investigation.

Table 23 is a summary for six modal analyses conducted on two filter substrates for three different work stations. We assumed that average particle volumes were similar for all mineral classes although coal particles tended to be larger. Weight percentages were calculated from the numerical counts using average specific gravities for each particle class. This approach is similar to that of Johnson et al (1981) applied to atmospheric particulate matter, except that they calculated particle volumes.

Point counts were conducted in sets of 100 as shown in Table 24 to give some statistical information as to reproducibility. There were no obvious trends in the point count sets which suggests that the standard deviations reflect sample heterogeneity rather than operator experience.

Table 23 MODAL ANALYSIS OF RESPIRABLE COAL DUSTS (1)

SAMPLE TYPE		GENERAL		SHEARER		BRUSHER	
SAMPLE/FILTER	S.G.	85-132 NUCLEPORE	85-4 MILLIPORE	85-175 NUCLEPORE	85-175(2) NUCLEPORE	85-174 NUCLEPORE	85-39 MILLIPORE
MINERAL		wt.(%)	wt.(%)	wt.(%)	wt.(%)	wt.(%)	wt.(%)
coal	1.30	46.86	45.32	53.74	43.92	25.25	36.52
illite	2.45	25.06	26.83	27.49	27.42	43.82	40.99
calcite	2.70	10.37	2.41	1.59	3.49	2.60	2.84
quartz	2.65	8.08	11.84	5.01	7.98	11.21	12.83
kaolinite	2.45	1.38	1.37	1.45	2.37	5.89	1.29
pyrite	5.00	1.13	0.00	2.95	8.61	0.96	1.58
siderite	3.80	0.86	0.85	0.90	2.45	6.58	1.20
other	2.91	6.25	11.38	6.87	3.76	3.64	2.76
TOTAL		100.00	100.00	100.00	100.00	99.94	100.00

1. Modal analysis based on 500 dust particle particles >lum randomly selected on moderatly loaded filter area. Particle recognition by EDI spectrum assigned to mineral class. All samples carbon coated except 85-132 (gold coated).

1. Wt. % from: $((A)_m*(SG_m)/\Sigma(A*SG_n))*100$

where:
A= percentage of particle count total
m= mineral of interest
SG= assigned specific gravity
n= mineral classes identified

note: particle volumes were assumed to be equivalent for all mineral classes

ABLE 24 RAW DATA FOR MODAL ANALYSIS

PARTICLE COUNTS - SAMPLE CBCRL 85-132

	1	2	3	4	5	AVERAGE (%)	STANDARD DEVIATION
coal	314	91	--	--	--	60.70	--
illite	92	27	--	--	--	17.89	--
calcite	34	15	--	--	--	8.21	--
quartz	27	4	--	--	--	4.00	--
kaolinite	5	4	--	--	--	1.78	--
pyrite	2	2	--	--	--	0.84	--
siderite	2	1	--	--	--	0.52	--
other	19	13	--	--	--	6.06	--
total	495	157				100.00	

PARTICLE COUNTS - SAMPLE CBCRL 85-4

	1	2	3	4	5	AVERAGE (%)	STANDARD DEVIATION
coal	66	64	67	54	61	62.40	4.67
illite	16	15	23	23	21	19.60	3.44
calcite	1	4	1	1	1	1.60	1.20
quartz	10	11	6	5	8	8.00	2.28
kaolinite	1	1	2	1	0	1.00	0.63
pyrite	0	0	0	0	0	0.00	0.00
siderite	0	0	0	1	1	0.40	0.49
other	6	5	1	15	8	7.00	4.60
total	100	100	100	100	100	100.00	

PARTICLE COUNTS - SAMPLE CBCRL 85-175

	1	2	3	4	5	AVERAGE	STANDARD DEVIATION
coal	69	73	79	60	70	70.00	6.18
illite	18	18	14	29	16	19.00	5.22
calcite	1	0	0	1	3	1.00	1.10
quartz	4	2	1	4	5	3.20	1.47
kaolinite	3	1	0	1	1	1.00	0.98
pyrite	0	1	1	2	1	1.00	0.63
siderite	0	1	0	1	0	0.40	0.49
other	5	4	5	2	4	4.00	1.10
total	100	100	100	100	100	99.60	

TABLE 24 (con't)

PARTICLE COUNTS - SAMPLE CBCRL 85-175(2)

	1	2	3	4	5	AVERAGE	STANDARD DEVIATION
coal	61	61	59	70	62	62.80	3.83
illite	25	21	25	12	21	20.80	4.75
calcite	0	4	2	3	3	2.40	1.36
quartz	10	4	3	4	7	5.60	2.58
kaolinite	0	3	2	2	2	1.80	0.98
pyrite	1	6	3	5	1	3.20	2.04
siderite	1	1	2	0	2	1.20	0.75
other	2	0	4	4	2	2.40	1.50
total	100	100	100	100	100	100.20	

PARTICLE COUNTS - SAMPLE CBCRL 85-174

	1	2	3	4	5	AVERAGE	STANDARD DEVIATION
coal	31	48	47	45	31	40.40	7.74
illite	51	25	30	39	41	37.20	9.04
calcite	3	1	1	2	3	2.00	0.89
quartz	5	7	13	6	13	8.80	3.49
kaolinite	4	8	6	3	4	5.00	1.79
pyrite	1	0	0	1	0	0.40	0.49
siderite	4	6	3	2	3	3.60	1.36
other	1	5	0	2	5	2.60	2.06
total	100	100	100	100	100	100.00	

PARTICLE COUNTS - SAMPLE CBCRL 85-39

	1	2	3	4	5	AVERAGE	STANDARD DEVIATION
coal	59	55	44	55	52	53.40	4.92
illite	22	29	41	31	36	31.80	6.43
calcite	3	0	2	3	2	2.00	1.10
quartz	11	10	10	9	6	9.20	1.72
kaolinite	2	0	2	0	1	1.00	0.89
pyrite	0	1	0	1	1	0.60	0.49
siderite	0	0	1	1	1	0.60	0.49
other	3	5	0	0	1	1.80	1.94
total	100	100	100	100	100	100.40	

Major components (>10%) were recognized in the same order of frequency for all 100 count sets except for the "other" category. Minor component and trace component counts were most affected by sample heterogeneity.

The summary, Table 23, shows that when calculated as weight percents, the dominant constituents of coal mine dust are coal and illite. Minor constituents quartz, calcite, pyrite, kaolinite, and siderite (?) fluctuate in abundance by work station and replicate analysis in response to either analytical variance or respective rock/coal/ stone dust contribution.

Replicate analyses on one Nuclepore filter (#85-175) shows large disparities for pyrite, siderite and coal abundance estimates. This may be attributed to any of the following:

1. Filter loadings: heterogeneous distribution of particles biased by the sample collection device as shown by concentric loadings.
2. Statistics: 500 counts are insufficient to differentiate 8 categories.
3. Spectral recognition: the "other" category accounted from 0 to 15% of counts (Table 24) which may be due to particle agglomeration or operator experience.
4. Particulate recognition: Millipore filters mask coal particles and show charging artifacts. This factor is considered minor because no clear difference is shown between data from Nuclepore or Millipore substrate.

Kelly et al (1980) have discussed conditions for fine particulate characterizations. To achieve an uncertainty of <10% in any category requires 100 counts. No single particle can account for more than a few percent of total area or weight. With our respirable dusts, coal generally accounts for >60% of particles or >40% by weight. In order to give 100 counts to minerals representing only 1% by volume would require 10,000 counts per filter. This requires automated SEM-EDX and image analysis.

FUSION METHOD FOR EDX ANALYSIS OF MAJOR ELEMENTS

INTRODUCTION

Quantitative major element analysis of silicate rocks by xray fluorescence spectrometry (XRF) requires sample preparation to remove particle size and interelement absorption effects. The standard procedures involve fusion techniques to convert the silicate rock to a glass, thereby removing heterogeneity and matrix effects. Hutchison (1974) summarized four fusion methods based on lithium tetraborate ($Li_2B_4O_7$) or $LiBO_2$ flux ratios (1.86:1, 5.4:1, 14:1, and 2:1) and fused at various temperatures (900°C, 1000°C, 1100°C).

Gulson and Lovering (1968) investigated the use of an electron microprobe to obtain whole rock major element analyses on such fused rock glass. Using a flux dilution of 5.5:1, they prepared glass disks, then coated with carbon, and analysed for 8 major elements by wavelength dispersive Xray at 10kv. Calibration curves were based on U.S.G.S. standard rocks and absorption or fluorescence corrections were omitted because of the dilution effect. Good correlation between microprobe, chemical and XRF data was obtained for seven oxides when ignition loss, i.e. volatiles, were accounted for. Oxides P2O5 and MnO were below detection (<0.25%) because count rates were low and accelerating voltages 10kv damaged the samples.

Subsequent studies have extended the usefulness of whole rock analysis by microprobe using either reduced flux (Wittkopp and O'Day 1973) or direct rock fusion (Rucklidge et al 1970, Brown 1977). Flux melting has the advantage of retaining P and S and has application to all rock compositions but suffers from reduced xray signal and errors in weighing.

Nicholls (1974) and subsequently Schimann and Smith (1980) published methods for whole rock analysis using direct fusion and energy dispersive xray microanalysis. Nicholls (1974) fused rock powder in air on an iridium strip heater at 1600-1800°C. Analysis for 10 elements was by Si(Li) detection, accelerating voltage of 15kv and 100 second counts. Data was corrected for background, overlap, and ZAF matrix corrections with results normalized free of volatiles and unanalysed minor elements. Standards consisted of single elements, oxides or standard rocks. Results showed a detection limit of 0.1 wt. % and problems with background and peak overlap for Na and P respectively. Otherwise, the results for major elements matched recommended values for standard rocks within acceptable deviations. Schimann and Smith (1980) also reported good results using an optical furnace for direct fusion and EDX for simultaneous analysis of up to 22 elements.

The application of EDX and fusion procedures to environmental dust samples has been investigated by Pella et al (1978). Using NBS SRM 1633 Fly Ash and SRM 1648 Particulate Matter, they prepared approximately 10:1 flux to sample dilutions fused at 1100°C. Twelve elements were detected using an energy dispersive xray fluorescence (EDXRF) system with a tungsten target operated at 20kv for 15 minutes to 1.5 hour counting intervals. This gave detection limits of 6 to 100ppm for most elements except silicon (1000 ppm). They conclude that acceptable accuracy can be obtained on environmental samples using lithium tetraborate fusion and EDXRF.

OUR STUDY

The foregoing literature survey invites the possibility of applying EDX analysis of fused rock to coal mine dust. The bulk of mine dust (coal, volatiles) would be removed by the fusion technique, but the residual coal minerals have chemistries similar to silicate rocks. Coal dust prepared as HT or LT ash could be characterized by major chemistry.

Our limited respirable dust sample size and limited project time did not allow for a full investigation of the various fusion procedures relative to coal dust. Instead, we elected to investigate a previously prepared XRF pellet prepared by lithium tetraborate fusion of HT coal ash.

The glass pellet was mounted in our SEM without coating and analysed for 50 seconds at 20 kv. EDX spectra for three pellet areas analysed in spot mode are documented in Appendix II 8a,b with corresponding ZAF corrected calculations.

Such a sample of glass conforms with thickness, homogeneity, and geometry requirements of the semi-quantitative standardless data processing package of Tracor Northern SSQ. As a result of fusion, the atomic number (Z) and fluorescence (F) factors approach unity. But absorption corrections (A) are excessive due to the chemical complexity of this material. Furthermore, the peak to background ratios are poor due to the flux dilution. The resultant calculations are sensitive to the selection of background regions especially for minor components.

Table 25 summarizes the EDX results for major oxides in comparison with whole rock XRF results. Correlation is poor and several major discrepancies are noted:

1. Minor elements up to 1.62% were not detected by EDX but reported by XRF.
2. Al is underestimated and Si overestimated by EDX relative to XRF. Since the XRF results were a single laboratory determination, an "accepted value" was not available.
3. Sulphur was calculated by the SSQ programme, up to 0.82 wt. % but was not apparent in the EDX spectra.

We conclude that analysis of fusion pellets is a promising technique but requires considerable further work. The following factors need investigation:

1. Increase counting statistics and background subtraction.
2. Loss of Na from beam damage or other volatile effects.
3. Application of standards.
4. Direct fusion methods applied to coal dust ash.
5. Preparation methods of polishing and coating fused pellets.

TABLE 25

X-RAY ANALYSIS OF FUSED COAL ASH
(SAMPLE 85-151-001)

		EDX		XRF	
ELEMENT	MAJOR OXIDE	ELEMENTAL wt. %	OXIDES wt. %	OXIDES wt. %	OXIDES (3) wt. %
Si	SiO2	55.89	63.74	54.60	53.35
Al	Al2O3	14.39	14.49	25.30	24.72
Ca	CaO	1.91	1.42	2.45	2.39
Mg	MgO	--	--	--	1.62
Na	Na2O	--	--	--	0.40
K	K2O	2.89	1.86	4.18	4.08
Fe	Fe2O3	24.25	18.49	12.24	11.96
Mn	MnO	--	--	--	0.11
Ti	TiO2	0.00	0.00	1.24	1.21
P	P2O5	--	--	--	0.15
Cr	Cr2O3	--	--	--	<0.01

note:
1. All data normalized to 100%
2. Fusion with $Li_2B_4O_7$ at 1000° C
3. XRF whole rock analysis reported by X-ray Analytical Laboratories, Don Mills Ontario

ORGANIC ELEMENTAL ANALYSIS

INTRODUCTION

The determination of carbon, hydrogen, nitrogen, and oxygen among other major elements is a critical analysis for coal, as indicated by the traditional analytical procedures (ASTM D3176, D3178, D3179, and D3180) and the industry term "ultimate analysis". Respirable coal dust samples are generally too small to allow application of standard ASTM tests. Nitrogen, for example, by ASTM D3179 requires a 1g of <250um (-60 mesh) sample. In contrast, modern instrumental analysers allow rapid multi-element microanalysis.

The Carlo Erba Elemental Analyser, model 1106, handles powder samples as small as 0.1mg to 10mg using a 4mm x 6mm tin or silver capsule. Belcher (1977) has described the instrument and analytical procedures in detail. The main disadvantages he identified were the lack of an ash determination and the influence of chlorine on oxygen determinations.

OUR STUDY

For our study, it was considered appropriate to compare coal dust filter collections from various work stations with whole coal values for C,H,N and O to reveal trends. The influence of chlorine on oxygen values was not considered to be a problem because of generally low chlorine in Sydney coals (<.27%). The ash values could be approximated by difference assuming C+H+N+O+ash = 100%.

Twelve heavily loaded personal sampler filters were selected representative of the various work stations. Five run-of-mine coals were analysed to represent the parent material, (Table 26).

For carbon, hydrogen, nitrogen, and oxygen analysis approximately 0.8mg of standards and samples were weighed into tin (for CHN) and silver (for O) capsules using a Mettler UM3 Ultramicrobalance. The samples were then loaded into the autosampler of the 1106 Carlo Erba Elemental Analyzer from which they dropped at preset times into a combustion tube.

CHN

The combustion tube for CHN analysis was maintained at 1030°C. The constant flow of helium through the tube was temporarily enriched with oxygen as each sample dropped for flash combustion. The combustion gases were reduced by passing them over copper at 650°C. The resulting gases were then separated in a Porapak QS chromatographic column maintained at 100°C into N2, CO2, and H2).

Oxygen

The combustion tube for oxygen analysis was maintained at 1060°C. A constant flow of helium carried the pyrolysis gases into a 5A Molecular Sieve chromatographic column maintained at 100°C which separated the gases into N2, CH4, H2, and CO.

At the end of the chromatographic column, a TCD detector produced an electrical signal proportional to the concentration of the gas. This signal was then passed on to a Spectra Physics 4270 integrator which calculated the area under the curve. The concentration was then calculated using the following equations.

1. K-FACTOR CALCULATION:
   The K-Factor was calculated for each element according to the following equation:

$$K = \frac{\%t \times WS}{1}$$

where:
$\%t$ = Theoretical percentage of the standard
Ws = Weight of the standard in mg
I = Standard integral

2. PERCENTAGE CALCULATION:
   The percentage of each element to be determined was calculated according to the following equation:

$$\% = \frac{K \times 1}{W}$$

where:
K = Average factor of each element
1 = Sample integral
W = Weight of sample in mg

Table 26 contains results for both coal and filter loadings.

Considerable variance of elemental values is apparent in both the R.O.M. coals and the work station dust collections. This is due to the heterogeneity of coal, the relative dilutions of coal, rock and stone dust as well as analytical variance.

Filter loadings collected at the longwall face appear to have higher carbon and nitrogen concentrations than the parent R.O.M. coal, with mean values as follows:

Source	**Mean %C SD**	**Mean %N SD**
dust at face:	69.0% ± 8.4	1.55% ± .29
R.O.M. coal:	61.7% ± 3.8	1.07% ± .14

ABLE 26

ELEMENTAL ANALYSIS DATA *

AMPLE TYPE	SAMPLE I.D.	CARBON (WT%)	HYDROGEN (WT%)	NITROGEN (WT%)	OXYGEN (WT%)	TOTAL	H/C**
ILTER SAMPLES							
BRUSHER	85-100	59.41	2.36	1.18	6.79	69.74	0.48
RUSHER	85-103	63.55	3.83	1.31	7.14	75.83	0.72
RUSHER	85-58	70.49	4.14	1.37	7.14	83.14	0.70
CONTROL PT.	85-81	68.09	4.20	1.44	6.18	79.91	0.74
FACE(5W)	85-72	72.25	4.69	1.36	6.44	84.74	0.78
FACE(5W)	85-73	59.60	3.93	1.19	7.10	71.82	0.79
ACE(5W)	85-83	78.01	4.97	2.05	6.03	91.06	0.76
FACE(9E)	85-104	73.30	4.39	1.52	6.60	85.81	0.72
ACE(9E)	85-63	73.35	4.54	1.53	7.05	86.47	0.74
ACE(9E)	85-97	57.36	3.79	1.64	6.88	69.67	0.79
SHEARER	85-34	75.60	4.52	1.36	10.27	91.75	0.72
SHOTFIRER	85-101	61.59	3.43	1.02	6.63	72.67	0.67
PARENT COAL							
LINGAN/HARBOUR (ROM)		61.58	4.05	1.18	6.26	73.07	0.79
NOVACO/HARBOUR W (ROM)		60.84	4.14	1.08	9.03	75.09	0.82
/HARBOUR E (ROM)		67.72	4.69	1.21	9.72	83.34	0.83
PRINCE/HUB (ROM)		57.16	3.88	0.88	8.50	70.42	0.81
ROGAN/LLOYD COVE (ROM)		61.14	4.10	0.99	9.78	76.01	0.80

* as received (ASTM D3180)
** atomic ratio

This can be explained by the reduced contribution of roof rock and stone dust to the dust levels during cutting of coal seams. If the parent coals were analysed on an "ash free, dry" basis, the mean %C and %N would be: 80± 4.6% and 1.39 ± .19% respectively. This compares with mean normalized %C and %N values for the filters of 84.57% and 1.90%.

The large standard deviations are partially due to changes in lithology during the mining operation and, therefore, changes in ash content of dust. Note that we have expressed all data on an "as received basis" (ASTM D3180 terminology) reflecting the moisture levels of the samples as arrived at the laboratory.

PROJECT SUMMARY

The foregoing chapters have documented the present state-of-the art in coal dust analysis. The results from SEM-EDX provide the most comprehensive suite of minerals for constituent analysis when applied to particle by particle point count procedures. The precision of this technique requires further documentation based on much larger statistical counts and replicate sample collections. The accuracy of the technique is at present difficult to assess because of the lack of alternate analytical methods. Furthermore, certain assumptions of particle volume and filter loadings need substantiation. Nevertheless, it is the authors' opinion that this point count technique by SEM-EDX holds the best promise of reliable routine multiconstituent analysis.

In contrast, it has been shown by Thakur, Riester and Parisi (1983) and others that current methods of single constituent analysis (ie. quartz) in respirable coal dust using XRD and I.R. are neither reproducable nor correlative. Regulatory requirements for respirable dust levels must take into consideration this present state of analytical limitations.

In addition to SEM-EDX, we recommend complementary chemical information by organic elemental and neutron activation analysis (INAA). Such an approach should reveal the mineral and chemical factors and conditions attributable to workers' pneumoconiosis. The Atlantic Coal Institute welcomes the opportunity to continue such a line of investigation.

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APPENDIX I - Part 1

CANADA DEPARTMENT OF LABOUR

STANDARD FOR THE CONTROL
OF
DUST CONCENTRATIONS IN
COAL MINES

Approved in accordance
with Subsection 10(b)
of the Canada Dangerous
Substances Regulations
for use in coal mines.

R.H. Elfstrom
R.H. Elfstrom,
Division Chief,
A.P.D.

2 July, 1975
Date

APC Branch
Ottawa, Ontario
July, 1975

CANADA DEPARTMENT OF LABOUR

STANDARDS FOR THE CONTROL OF RESPIRABLE DUST CONCENTRATION IN COAL MINES

Maximum Allowable Concentrations

1. The average dust concentration to which each individual is exposed shall not exceed (i) 4.3 mg/m^3 of respirable dust
(ii) 2 mg/m^3 of ash, and
(iii) 0.15 mg/m^3 of free silica
based on personal sampling on a portal-to-portal basis.

2. The average dust concentration shall be determined by a method that:

(i) meets the requirements of this standard; and
(ii) is acceptable to the Division Chief.

Excursion Factor

3. Excursions up to double the values of average concentrations specified in Section 1 are permitted for periods of up to one full shift as long as they are compensated by exposures to dust concentrations below the specified value averaged out during the course of each 40 hour work week. This requires that the total mass of dust calculated by multiplying the dust concentration by the time of exposure in hours does not exceed the value obtained by multiplying the specified value of 4.3 mg/m^3 by 40 for each regular 40 hour work week.

Control of Longwall Coal Faces

4. The exposure of face workers on longwall coal faces may, instead of using personal samplers, be monitored by measurements of the mass concentration of respirable dust in the return airway. An average of 4.3 mg/m^3 exposure for all workers as measured by personal sampling, will be met if the mass concentration of respirable dust measured at a point 70 metres from the face in the return airway does not exceed a maximum value of 8 mg/m^3 when measured in accordance with this standard.

Approval of Sampling for Longwall Faces

5. Approval for sampling in the return airway of longwall faces in lieu of personal sampling of all face workers will be given when acceptable dust levels are obtained. The conditions for approval are that two consecutive samples of a series taken during coal production on alternate days are below 8 mg/m^3 total respirable dust.

FOREWORD

Control of Airborne Contaminants

Section 10 of the Canada Dangerous Substances Regulations requires the employer to ensure that the ventilation of any dangerous substance that may be carried in the air where an employee is working:

(a) does not exceed the threshold limit value recommended by the ACGIH, or

(b) conforms with any standard that

  (i) follows good industrial safety practice, and

  (ii) is acceptable to the Division Chief.

Acceptable standards for concentration of airborne contaminants in coal mines must be such that personal exposure of miners over a working life will not result in new incidences of pneumoconiosis or the further development of such disease in any person.

As the first step towards the goal of zero risk of pneumoconiosis, the current U.K. Standards have been adopted for application in Cape Breton Coal Mines. These standards require that the mean average lifetime exposure of coal face workers while at work shall not exceed 4.3 mg/cu^3 of respirable dust. To control the hazards from free silica and other respirable particles, the respirable dust shall not contain more than 2 mg/cu^3 of ash nor more than 0.15 mg/cu^3 of free silica.

The U.K. standard is based is based on research results which indicate that a 4.3 mg/cu^3 average exposure level would give a 3.4% maximum probability of contracting simple pneumoconiosis after 35 years of exposure. In order to reach the goal of zero risk, the standard will be progressively lowered to 3 mg/cu^3, at which level the risk is estimated at 1%, and then to the zero risk level of 2 mg/cu^3.

In order to ensure uniform interpretations and thereby facilitate the application of the regulations, a standard has been prepared giving details of acceptable levels of contaminants, sampling procedures and methods of measurement, together with the limits of extent and duration of excursions of dust concentration that will be permitted under the provisions of Sections 10 and 11 of the Canada Dangerous Substances Regulations.

6. When the control of respirable dust during work on a longwall face is acceptable as demonstrated in (5) above, monthly sampling will be authorized by the Mine Safety Engineer and may be continued so long as the measured concentration does not exceed 8 mg/m^3. Whenever a monthly sample shows a concentration exceeding 8 mg/m^3 the control of dust on the longwall face is no longer considered acceptable. Action must immediately be taken to reduce the concentration and sampling must be carried out on alternate days until the control of dust at the face is again acceptable.

Operation at Above-standard Concentrations

7. Since 8 mg/m^3 is the maximum concentration permitted in the return airway to maintain the personal average exposure at 4.3 mg/m^3, no regular operation will be permitted if the dust concentrations measured in accordance with this standard have exceeded 8 mg/m^3 averaged over one month, except in accordance with the requirements of Section 8.

8. If a longwall face is not approved for monitoring by measuring dust concentration in the return airway, personal sampling of the high risk worker at the coal face must be carried out weekly in addition to the sampling in the return airway that is being done to obtain approval of the workplace. For continued operations, the average of the last three personal samples must not exceed 4.3 mg/m^3 total dust.

9. If the conditions in Section 8 cannot be met, operations must be discontinued until corrective measures are taken. Operations may then be carried out for one week at a time to permit testing of the dust concentrations.

Application for Exemption From Standard

10. Application for exemption from the requirement of this standard shall be made in writing to the Division Chief. Any such application shall contain the following information:

(i) Identification of the working place and mine for which exemption is requested.

(ii) Results of dust surveys and record of action taken to reduce dust concentrations.

(iii) Description of ventilation system of mine and working place involved and data on quantity and velocity of air at workplace.

(iv) Water volume and pressure at workplace and type, number and location of any water sprays in use.

(v) Statement by ventilation or dust control engineer of action proposed to meet the standard and the estimated time for completion.

Requirements for Sampling and Testing of the Atmosphere

11. The Canada Dangerous Substances Regulations require that the employer sample and test the atmosphere of any area in which an employee is working when the area is subject to contamination by a dangerous substance. The sampling frequency required under these regulations shall be as follows:

(i) Longwall faces for coal face production workers

Initial sampling frequency & conditions for approval of workplace : Samples to be taken during coal production on alternate days until two consecutive readings are below 8 mg/m^3 total respirable dust.

Sampling frequency when longwall is approved : Monthly.

Sampling location : 70 metres from face in return airway.

Sampling period : While men are at workface: does not include travelling time.

When concentration exceeds 8 mg/m^3 sampling shall be continued on alternate days during the period that remedial action is being taken to reduce the concentration until two consecutive readings below 8 mg/m^3 are recorded. Since dust control work may be needed to reduce the concentration, sampling may be deferred for one week to allow for corrective work to be done. Monthly sampling will be recommended when the face is again acceptable.

(ii) Stone drivages and other operations where quartz content of dust exceeds 5%

Sampling location : personal sampler carried by worker or fixed sampler set within 3 feet of worker.

Sampling frequency : one per month for each working place; once every two months for the high risk man; once per year for all other workers.

Sampling period : portal to portal

Acceptable conditions : the mean of the last three measured concentrations shall not exceed 6 mg/m^3 total dust, 3 mg/m^3 ash and 0.15 mg/m^3 free silica.

When the mean exceeds any of the limits, sampling shall be continued on alternate days until the average of the last three readings meets all three requirements. In the case of annual samples for other workers, if the sample exceeds the standard, samples shall be taken monthly on that man until 2 consecutive samples are within limits.

(iii) All other operations

Sampling location : Personal sampler work by worker

Sampling frequency : Annually for each person

Sampling period : Portal to portal

Acceptable conditions : Sample shall not exceed 6 mg/m^3 total dust, 3 mg/m^3 ash and 0.15 mg/m^3 free silica.

When concentrations exceed the limits specified samples shall be taken monthly until 2 consecutive samples are within limits. If other high-risk operations are located the frequency of sampling will be determined by the Mine Safety Engineer on the basis of the measured concentrations.

Whenever an individual sample varies from previous samples by more than a factor of 3, the sample should be checked to determine the cause of the variation and a further sample should be taken if no cause is apparent.

Methods of Assessment of Airborne Dust

12. Total Respirable Dust shall be determined by weighing on a balance with an accuracy of 0.03 mg.

The ash content of respirable dust is to be determined by weighing before and after ashing to a constant weight.

The respirable quartz content of samples shall, until further notice, be determined by the Elliott Lake Laboratory of EMR using X-ray diffraction or by other laboratories using procedures approved by the Division Chief.

The employer shall take samples and have quartz determinations done at the time of opening of each new section and any significant change of strata and whenever the ash content of samples is such as to lead to the expectation that the quartz content could exceed 0.06 mg/m^3. Quartz analysis shall be done in all samples where the quartz content is expected to exceed 30%.

Equipment

13. The standard instrument shall be the Cassella 113A. Other instruments will be acceptable if they have been calibrated by an approved method to give comparable results to the Casella 113A.

The determination of the free silica content of samples by X-ray diffraction or by infra-red spectroscopy is acceptable.

Records

14. The company shall maintain records of each individual miner showing occupation, places worked, and dust exposures. Those records shall be correlated with medical records to provide a basis for verification or modification of the exposure standards.

Future standards

15. The target for dust control should be to obtain concentrations of respirable coal dust of not more than 4 mg/m^3 measured in the return airway of longwall faces in order to limit the exposure of individual miners to the average level of 2 mg/m^3 that is considered by the National Coal Board to represent a zero risk of pneumoconiosis.

APC Branch
Ottawa, Ontario.
June, 1975.

APPENDIX I - Part 2

70. (1) The mine manager shall ensure that coal dust is not allowed to accumulate in any place in a mine, that dry areas that produce coal dust are systematically wetted down with water and that other dust control measures acceptable to the Chief Inspector are employed to reduce the risk of a coal dust explosion in such areas.

(2) To reduce coal dust created by coal cutting machines operating at a working face that is not naturally wet, a jet of water shall be directed over the pick chain where it enters the cut.

(3) Unless mined coal is naturally wet, it shall be sprayed with water before it is loaded with a coal loading machine, dumped into mine cars or subjected to any handling that will create dust.

71. (1) Unless exempted in writing by the Chief Inspector, every roadway in a mine shall be treated with stone dust or other non-combustible material in such manner and at such intervals as are approved by the Chief Inspector to ensure that the dust on the floor, roof and sides of every roadway contains at least 65 per cent stone dust or other non-combustible material, but where methane gas is present in the air over a roadway additional stone dust or other non-combustible material shall be applied to ensure that for every one per cent of methane gas present in the air the percentage of stone dust or other non-combustible material on the floor, roof and sides of the roadway is increased by 10 per cent from the base of 65 per cent.

(2) Unless a lesser quantity is approved by a safety officer, not less than 20 bags of stone dust shall be stored for emergency use within a reasonable distance of each working face in each working section of a mine.

(3) Stone dust or other non-combustible material used for the purpose referred to in subsection (1) shall contain not less than 70 per cent by weight of fine material that is capable, when dry, of passing through a sieve of 200 mesh.

(4) Where a larger percentage of stone dust or other non-combustible material is used than is required by subsection (1), the percentage of fine material referred to in subsection (3) may be reduced in proportion to the increase in the amount of non-combustible dust used in excess of the minimum required, but in no case shall the percentage of fine material be less than 25 per cent.

(5) At intervals of not less than once in every month dust samples shall be taken from roadways in a mine by a competent person appointed by the mine manager and such samples shall be taken in accordance with a sampling plan approved in writing by the Chief Inspector or a safety officer and tested for combustible material by a method approved in writing by the Chief Inspector.

1978

CHAPTER 1011

CANADA LABOUR CODE

Coal Mines (CBDC) Safety Regulations

REGULATIONS RESPECTING SAFE EMPLOYMENT IN CAPE BRETON DEVELOPMENT CORPORATION COAL MINES

18. Subsection 71(1) of the said Regulations is revoked and the following substituted therefor:

"71. (1) Unless exempted in writing by the Chief Inspector, every roadway in a mine shall be treated with stone dust or other non-combustible material in such a manner and at such intervals as are approved by the Chief Inspector to ensure that the dust on the floor, roof and sides of every roadway contains at least 75 per cent stone dust or other non-combustible material, but where flammable gas is present in the air over a roadway, additional stone dust or other non-combustible material shall be applied to ensure that for every one per cent of flammable gas present in the air the percentage of stone dust or other non-combustible material on the floor, roof and sides of the roadway is increased by 5 per cent from the base of 75 per cent.

Amended: July 3, 1981:

Coal Mines Regulation Act

1972

CHAPTER 36

REVISED STATUTES
of
NOVA SCOTIA 1967

QUEEN'S PRINTER
Halifax, Nova Scotia

PRECAUTIONS AGAINST COAL DUST

69 (1) Unless the floor, roof and sides of the road and working places in a mine are naturally wet throughout, they shall be systematically cleared so as to prevent, as far as practicable, the accumulation of coal dust, and systematic steps either by way of watering or otherwise, shall be taken to prevent explosions of coal dust occurring.

(2) When machines are working at the coal face they shall be equipped so that,

(a) when continuous miner machines are mining coal suitable sprays of water, or water and some suitable additive, shall be so arranged as to allay the coal dust created by such machines;

(b) except in working places that are naturally wet, coal cutting machines while cutting coal shall have a jet of water, or water and some suitable additive, directed over the pick chain where it enters the cut;

(c) except in working places that are naturally wet, coal loading machines shall have the pile of coal sufficiently wetted with water, or water and some suitable additive, before commencing to load;

(d) water sprays or jets shall be used where coal is delivered from conveyors into mine cars and on landings before coal is placed on the rope for hoisting, and such other places as may be required by an Inspector [inspector]. In cases where coal is already wet, this does not apply.

(3) The floor, roof and sides of every road or part of a road, that is accessible, shall, if deemed necessary by an inspector, be treated in one of the following ways,

(a) they shall be treated with incombustible dust in such manner and at such intervals as will ensure that the dust on the floor, roof and sides, respectively, shall always consist throughout of a mixture containing not more than thirty-five per cent combustible matter, or such other greater or less percentage as an inspector upon investigation may deem proper. Where methane gas is present, the percentage of incombustible dust shall be increased by ten per cent for each one per cent of methane in the air current, as determined by analysis of an air sample from the section; or

(b) they shall be treated in such other manner as the Minister may approve.

(4) The obligation imposed by subsection (3) shall not apply in respect to any road, or part of a road, if and so long as the natural conditions thereof, as regards presence of incombustible dust and moisture are found by tests, made in accordance with this Act, to be such as to comply with the foregoing requirements.

(5) Not less than twenty bags of stone dust shall be stored in every working section for emergency within a reasonable distance of the working face. In room and pillar sections, a suitable amount of stonedust [stone dust] shall be kept within easy access of the working faces.

(6) The incombustible dust used for the purpose of subsection (3) shall contain not less than fifty per cent by weight of fine material, capable, when dry, of passing through a sieve of two hundred mesh to the lineal inch (forty thousand to the square inch) but, if a larger portion of incombustible dust is used than is required by subsection (3), the percentage of fine materials contained in the incombustible dust may be reduced proportionately, but shall not be less than twenty-five per cent.

(7) For the purpose of testing the composition of the dust mixture in any part of a road, the following procedure shall be adopted,

(a) Representative samples of the dust shall be collected from the floor, roof and sides, respectively, over an area of road not less than fifty yards in length, and shall comprise the dust collected on the roof and sides and to a depth not exceeding one quarter of an inch on the floor.

(b) Each sample collected shall be well mixed and a portion of the mixture shall be sieved through a piece of metallic gauze, having a mesh of twenty-eight to the lineal inch.

(c) A weighed quantity of the dust which has passed through the sieve shall be dried at two hundred and twelve degrees Fahrenheit, and the weight lost shall be reckoned as moisture. The sample shall then be brought to red heat in an open vessel until it no longer loses weight. The weight so lost shall be reckoned as combustible matter for the purpose of the test.

(d) In dust mixtures which contain carbonates:

(i) a weighed quantity of the dried dust shall be heated in an open vessel to a temperature high enough to ensure complete decomposition of the carbonates (the high temperature of a blowpipe flame is required, and heating continued for about an hour) and until it no longer loses weight; and the percentage loss of weight shall be determined;

(ii) a weighed quantity of the dried dust shall be treated with dilute acid in a suitable apparatus and the percentage loss of weight due to the evolution of carbonic gas shall be determined;

(iii) the difference between the two percentage losses of weight so determined shall be reckoned as the percentage of combustible matter for the purpose of the test.

(8) Tests of samples of dust, so taken as to be representative of the normal composition of the dust throughout the roads of the mine on the floor, roof and sides, respectively, shall be made as often as the inspector deems necessary, and the result of

these tests shall be recorded and a copy sent to the deputy inspector of the district.

(9) No dust of a kind that is prohibited by the Minister on the ground that it would be injurious to the health of the persons working in the mine shall be used for the purpose of complying with this Section, but if any dispute arises as to whether the dust is injurious, it shall be determined in the manner provided by Section 63. R.S., c. 36, s. 69.

VENTILATION

70 (1) Every mine while being worked shall be thoroughly ventilated and furnished with an adequate supply of pure air to dilute and render harmless inflammable and noxious gases to such an extent that all underground places, except places sealed or properly fenced off according to law, shall be in a safe and healthy condition for the persons passing and working therein.

(2) An adequate supply of pure air shall mean such volume of air circulating in a mine and the working places thereof as may be deemed necessary by the deputy inspector for the district in which the mine is situated.

(3) Main fans shall be located on the surface in fireproof housing or casing offset from the line of any mine opening. The housing, casing or drifts shall be provided with ample pressure, relief doors or other devices easily opened by the force of an explosion. The installation shall permit prompt reversal of the air flow.

(4) When the fan is stopped for any reason, the workmen shall be removed from their working places to a place of safety and, if the period of stoppage exceeds a period of time to be decided upon by the manager and the inspector concerned, the workmen shall be removed to the surface. When workmen have been removed from their working places for reason of the fan stopping, the mine shall be examined by some competent person or persons and the workmen shall not return to their working places until the mine has been declared safe.

(5) Every mine shall be divided into such number of districts or splits as in the opinion of an inspector will furnish each district or split with the greatest possible quantity of fresh air.

(6) All intake air shall travel free from all stagnant water, old workings and other places likely to contaminate the air, and the return air from each district or split shall, if necessary, be conducted by an overcast or an undercast into the return airway, which shall lead to the main outlet.

(7) In every working place in a mine where the current of air is not sufficient to keep the place clear of inflammable or noxious gases, brattice or some other method approved by an inspector shall be used to conduct the air to the working face.

(8) A booster fan may be installed or operated in any mine only on the written permission of an inspector, and such fan after it is installed shall be run continuously. At the completion of each shift a report on the condition of each fan shall be entered in a book kept at the mine for that purpose.

(9) An auxiliary fan may be installed or operated in a mine only on the written permission of an inspector, and after such fan has been installed,

(a) it shall be run continuously except with the permission of an inspector first obtained;

(b) a sufficient quantity of air shall at all times reach it so as to ensure that recirculation does not take place;

(c) it shall use not more than forty per cent of the air passing the fan;

(d) it shall be situated on the intake side and at least twenty feet out by the last open cross-cut or entrance to the place being ventilated;

(e) it shall have air ducts or tubing for conducting the air to the place being ventilated and such air ducts or tubing shall be maintained in such condition as to minimize air leakage and to ensure an adequate supply of air being delivered within fifteen feet of the face;

(f) if the fan is electrically driven the motor and switch shall be permissible;

APPENDIX I - Part 4

TABLE 4

ATLANTIC COAL INSTITUTE PROJECT # 84-129

SAMPLE LOG

SAMPLE NUMBER	DATE REC'D	FILTER TYPE	SAMPLING LOCATION	ANALYSIS SEM-EDI	INNA	ELEMENTAL	PARTICLE LOADINGS
CBDC-1	DEC. /85	GLASS FIBRE			x		ZONED
CBDC-2	DEC. /85	GLASS FIBRE			x		ZONED
CBDC-3	DEC. /85	GLASS FIBRE			x		ZONED
CBDC-4	DEC. /85	GLASS FIBRE			x		ZONED
CBDC-5	DEC. /85	GLASS FIBRE			x		ZONED
CBDC-6	DEC. /85	GLASS FIBRE					ZONED
CBDC-7	DEC. /85	GLASS FIBRE					ZONED
J. HWANG	DEC. 28/85	PVC					ZONED
J. HWANG	DEC. 28/85	MILLIPORE(?)					
J. HWANG	FEB. 5/85						
J. HWANG	FEB. 5/85						
J-1 OPEN FACE	JAN. 10/85						ZONED DISLODGED
MSA-1 C-13	JAN. 10/85						ZONED
MSA-2 C-14	JAN. 10/85						ZONED
MSA-3 C-15	JAN. 10/85						ZONED
CBCRL #85- 1	FEB. 18/85	MILLIPORE	GENERAL				ZONED
CBCRL #85- 2	FEB. 18/85	MILLIPORE	COAL FACE	x			ZONED
CBCRL #85- 3	FEB. 18/85	MILLIPORE	COAL FACE				ZONED
CBCRL #85- 4	FEB. 18/85	MILLIPORE	COAL FACE	x	x		ZONED
CBCRL #85- 5	FEB. 18/85	MILLIPORE	COAL FACE		x		ZONED DISLODGED
CBCRL #85- 6	FEB. 18/85	MILLIPORE	COAL FACE				ZONED
CBCRL #85- 7	FEB. 18/85	MILLIPORE	GENERAL		x		ZONED
CBCRL #85- 8	FEB. 18/85	MILLIPORE	CONTROL POINT		x		ZONED
CBCRL #85- 9	FEB. 18/85	MILLIPORE	BRUSHING (TOP)	x	x		ZONED
CBCRL #85- 10	FEB. 18/85	MILLIPORE	BRUSHING (TOP)				ZONED
CBCRL #85- 11	FEB. 18/85	MILLIPORE	BRUSHING (TOP)				ZONED
CBCRL #85- 12	FEB. 18/85	MILLIPORE	GENERAL				ZONED
CBCRL #85- 13	FEB. 18/85	MILLIPORE	COAL FACE				ZONED
CBCRL #85- 14	FEB. 18/85	MILLIPORE	BRUSHING (BOT)				ZONED
CBCRL #85- 15	JAN. 21/85	NUCLEPORE		x			
CBCRL #85- 22	JAN. 21/85	NUCLEPORE		x			
CBCRL #85- 23	JAN. 21/85	NUCLEPORE		x			
CBCRL #85- 31	FEB. 18/85	MILLIPORE			x		ZONED
CBCRL #85- 32	FEB. 18/85	MILLIPORE	COAL FACE				ZONED
CBCRL #85- 33	FEB. 18/85	MILLIPORE	COAL FACE				ZONED
CBCRL #85- 34	FEB. 18/85	MILLIPORE	COAL FACE	x		x	ZONED DISLODGED
CBCRL #85- 35	FEB. 18/85	MILLIPORE	COAL FACE		x		ZONED DISLODGED
CBCRL #85- 36	FEB. 18/85	MILLIPORE	COAL FACE				ZONED DISLODGED
CBCRL #85- 37	FEB. 18/85	MILLIPORE	COAL FACE				ZONED
CBCRL #85- 38	FEB. 18/85	MILLIPORE	BRUSHING (TOP)	x	x		ZONED DISLODGED
CBCRL #85- 39	FEB. 18/85	MILLIPORE	BRUSHING (TOP)	x	x		ZONED
CBCRL #85- 40	FEB. 18/85	MILLIPORE	GENERAL		x		ZONED
CBCRL #85- 41	FEB. 18/85	MILLIPORE	BRUSHING (TOP)				ZONED DISLODGED
CBCRL #85- 42	FEB. 18/85	MILLIPORE	CONTROL POINT				ZONED DISLODGED
CBCRL #85- 43	FEB. 18/85	MILLIPORE	BRUSHING (BOT)				ZONED
CBCRL #85- 44	FEB. 18/85	MILLIPORE	COAL FACE				ZONED DISLODGED

TABLE 4 (con't)

CBCRL #85- 44	FEB. 18/85	MILLIPORE	COAL FACE				ZONED DISLODGED
CBCRL #85- 45	FEB. 18/85	NUCLEPORE	CONTROL POINT				
CBCRL #85- 46	FEB. 7/85	MILLIPORE					IN CASSETTES
CBCRL #85- 47	FEB. 18/85	MILLIPORE					IN CASSETTES
CBCRL #85- 48	FEB. 18/85	MILLIPORE					IN CASSETTES
CBCRL #85- 49	FEB. 18/85	MILLIPORE	BRUSHING (BOT)				ZONED DISLODGED
CBCRL #85- 50	FEB. 18/85	MILLIPORE	COAL FACE				ZONED DISLODGED
CBCRL #85- 51	FEB. 18/85	MILLIPORE	COAL FACE				ZONED DISLODGED
CBCRL #85- 52	FEB. 18/85	MILLIPORE	COAL FACE		x		ZONED DISLODGED
CBCRL #85- 53	FEB. 18/85	MILLIPORE	COAL FACE	x	x		ZONED DISLODGED
CBCRL #85- 54	FEB. 18/85	MILLIPORE	COAL FACE				ZONED DISLODGED
CBCRL #85- 55	FEB. 18/85	MILLIPORE	BRUSHING (TOP)				ZONED DISLODGED
CBCRL #85- 57	FEB. 18/85	MILLIPORE	GENERAL				
CBCRL #85- 58	FEB. 18/85	MILLIPORE	BRUSHING (TOP)			x	ZONED DISLODGED
CBCRL #85- 60	FEB. 18/85	NUCLEPORE	CONTROL POINT				
CBCRL #85- 62	FEB. 18/85	MILLIPORE	BRUSHING (BOT)		x		ZONED
CBCRL #85- 63	FEB. 18/85	MILLIPORE	COAL FACE			x	ZONED DISLODGED
CBCRL #85- 70	FEB. 18/85	MILLIPORE	GENERAL				ZONED
CBCRL #85- 72	FEB. 18/85	MILLIPORE	COAL FACE	x		x	ZONED DISLODGED
CBCRL #85- 73	FEB. 18/85	MILLIPORE	COAL FACE		x	x	ZONED DISLODGED
CBCRL #85- 74	FEB. 18/85	MILLIPORE	COAL FACE		x		ZONED
CBCRL #85- 75	FEB. 18/85	MILLIPORE	COAL FACE				ZONED DISLODGED
CBCRL #85- 76	FEB. 18/85	MILLIPORE	BRUSHING (TOP)	x	x		ZONED DISLODGED
CBCRL #85- 77	FEB. 18/85	MILLIPORE	COAL FACE				ZONED DISLODGED
CBCRL #85- 79	FEB. 18/85	MILLIPORE	BRUSHING (TOP)				ZONED DISLODGED
CBCRL #85- 81	FEB. 18/85	NUCLEPORE	CONTROL POINT			x	
CBCRL #85- 83	FEB. 18/85	NUCLEPORE	COAL FACE			x	
CBCRL #85- 85	FEB. 18/85	MILLIPORE	BRUSHING (BOT)				ZONED
CBCRL #85- 95	FEB. 18/85	MILLIPORE	COAL FACE				ZONED DISLODGED
CBCRL #85- 96	FEB. 18/85	MILLIPORE	COAL FACE		x		ZONED DISLODGED
CBCRL #85- 97	FEB. 18/85	MILLIPORE	COAL FACE			x	ZONED DISLODGED
CBCRL #85- 98	FEB. 18/85	MILLIPORE	COAL FACE	x	x		ZONED DISLODGED
CBCRL #85- 99	FEB. 18/85	MILLIPORE	BRUSHING (TOP)				ZONED DISLODGED
CBCRL #85- 100	FEB. 18/85	MILLIPORE	BRUSHING (TOP)			x	ZONED DISLODGED
CBCRL #85- 101	FEB. 18/85	MILLIPORE	BRUSHING (TOP)			x	ZONED DISLODGED
CBCRL #85- 102	FEB. 18/85	MILLIPORE	COAL FACE				ZONED DISLODGED
CBCRL #85- 103	FEB. 18/85	MILLIPORE	BRUSHING (BOT)			x	ZONED DISLODGED
CBCRL #85- 104	FEB. 18/85	MILLIPORE	COAL FACE			x	ZONED DISLODGED
CBCRL #85- 105	FEB. 18/85	MILLIPORE	BRUSHING (BOT)				ZONED DISLODGED
CBCRL #85- 107	FEB. 14/85	MILLIPORE					ZONED DISLODGED
CBCRL #85- 108	FEB. 14/85	MILLIPORE					ZONED
CBCRL #85- 109	FEB. 14/85	MILLIPORE					ZONED
CBCRL #85- 111	FEB. 14/85	NUCLEPORE					ZONED DISLODGED
CBCRL #85- 112	FEB. 14/85	NUCLEPORE					ZONED DISLODGED
CBCRL #85- 113	FEB. 14/85	NUCLEPORE					
CBCRL #85- 114	FEB. 18/85	PVC	CONTROL POINT				
CBCRL #85- 118	FEB. 18/85	GLASS FIBRE					ZONED DISLODGED
CBCRL #85- 131	FEB. 11/85	MILLIPORE	GENERAL				ZONED
CBCRL #85- 132	FEB. 11/85	NUCLEPORE	GENERAL	x			ZONED
CBCRL #85- 174	MAR. 4/85	NUCLEPORE	BRUSHING (TOP)	x			ZONED
CBCRL #85- 175	MAR. 4/85	NUCLEPORE	COAL FACE	x			ZONED

1.a PHOTOMICROGRAPHS: Coal Minerals

1. Lingan-Harbour coal ashed by low temperature oxygen plasma (LTA). This fragment shows minerals as pseudomorphic castes with voids representing maceral sites. Pyrite crystallites form a lenticular pod. x1500 Secondary electron image (SEI) coated with gold (Au).

2. Enlarged coal LTA photo #1, showing illite flake (EDX spectrum #2). Note that clays form flakes down to submicron dimensions. x15000 (SEI), Au coated. Crosshairs mark EDX spot analysis.

3. Lingan-Harbour LTA showing amorphous flocculated material. Only pyrite octahedra and framboids are recognizable. x1500 (SEI), Au coated.

4. Pyrite octahedron from Minto-Chipman #1 coal L.T.A.. Note the adhered flakes of clays and amorphous material. Backscattered electron image (B.E.I.), Au coated.

5. Massive pyrite from Lingan-Harbour LTA, X 1500 (BEI).

6. Pyrite framboids in Lingan-Harbour LTA. Note that framboids are composed of equant crystals of respirable dust size. Under oxidizing conditions the framboids disintegrate. x3500 (SEI), Au coat. The LTA has been treated with dilute HCl to remove flocculates.

7. Pyrite framboid in Lingan-Harbour LTA. As for photo #6, these crystal colonies are essentially composed of respirable dust size octabedra averaging 1 micron in diameter. LTA treated with HCl To remove flocculates. x7500 (SEI), Au coated.

8. Enlargement of Lingan-Harbour LTA pyrite framboid showing variance in crystallite morphology and dimensions. LTA treated with HCl to remove flocculates. Au coated, x10,000 (SEI).

9. Polished coal pellet of Minto-Chipman #1 X 35 (BEI). The bright grains are higher mean atomic number, dominantly pyrite (Fe). Carbon coated (C).

10. Coal Pellet: Minto-Chipman #1 - x2000 (BEI) showing a pyrite (1) framboid colony with kaolinite infill (2). Note the different degrees of cementation of the respirable dust size crystallites. x2000 (BEI) carbon coated.

11. Coal pellet: Minto-Chipman #1 carbominerite grain. Black layers are coal macerals. Grey grains are quartz, illite and pyrite. x1500 (BEI), carbon coated.

12. Coal pellet: Novaco-Harbour E, - x750 showing massive pyrite. The inclusion within pyrite above the crosshairs is a Ni bearing mineral. Most trace elements can be assigned to such micron size minerals. Carbon coated, BEI.

13. Coal pellet: Minto/Chipman #1 - x1000 (SEI). Pyrite octahedra forming a "chain" as cleat fillings.

14. Enlargement of crystals from photo #13., Although this is a polished pellet, these recessed crystals have retained their 3 dimensional morphology. To test the role of sample geometry we have conducted EDX spot analyses on individual crystal faces - x 3500 (BEI), carbon coated.

15. Coal pellet: Brogan-Lloyd Cove: x 15000, (SEI), carbon coated. These blocky crystals of pyrite illustrate the less common elongate habit down to submicron dimensions.

16. Coal pellet: Minto-Chipman #1, coated, x 3500 (BEI). Pyrite framboids showing internal crystals and overgrowth.
The degree of cementation by secondary pyrite can dictate the particle size.

17. Coal pellet: Drummond-Acadia x 350 (BEI), carbon coated. Carbominerite showing large anhedral quartz grains and bright pyrite crystals and framboids.

18. Coal pellet: Evans #5 x 5000 (SEI), carbon coated. Detrital zircon (1) grain associated with clays (3) and macerals (2) but likely derived from roof rock.

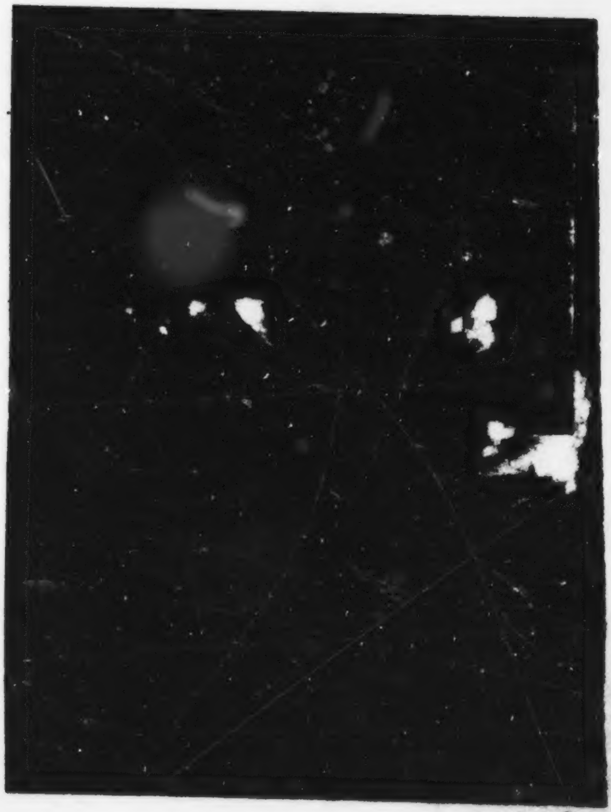
19. Coal fracture surface: Minto-Chipman #1 showing maceral morphology. We expect coal to contribute a significant number of elongate fragments to respirable dust. x 3500 (SEI), gold coated.

20. Weathering product on pyrite LTA: likely hematite. x 10000 (SEI). Such oxidation products could contribute to the respirable dust.

1

2

3



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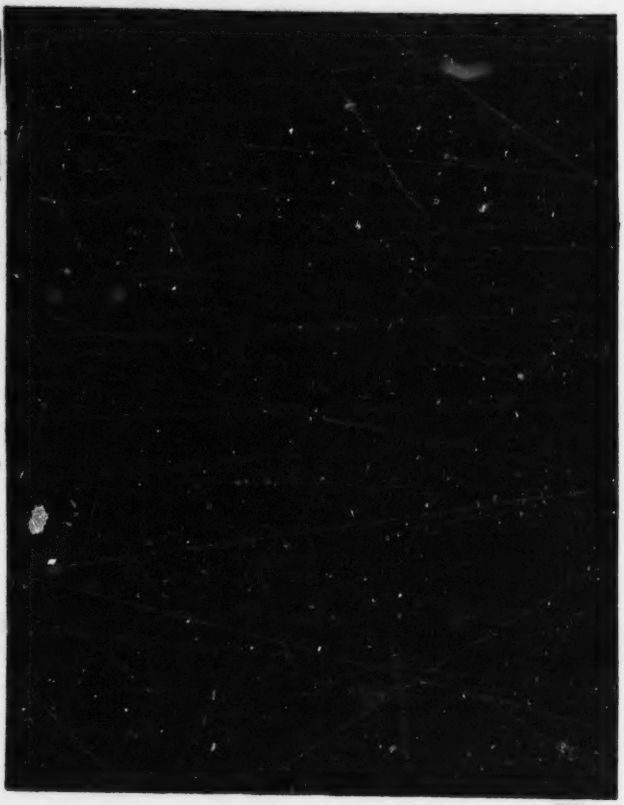

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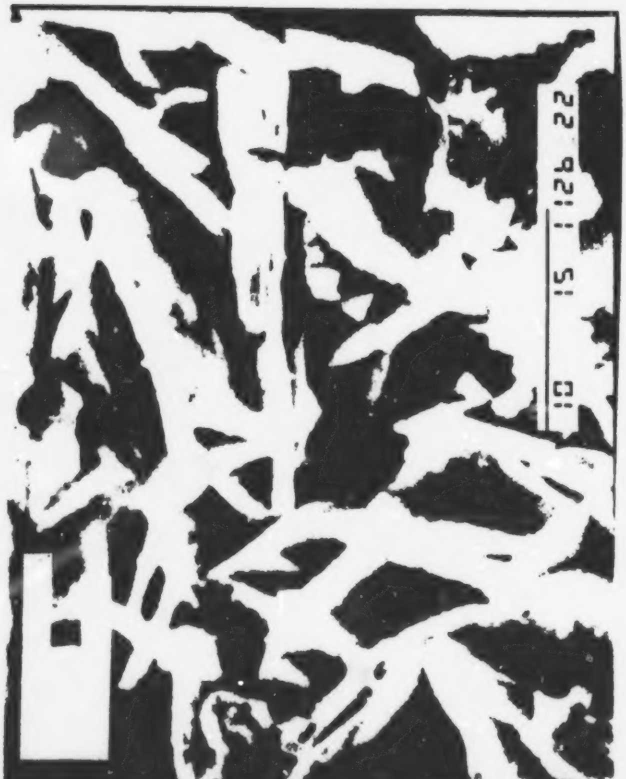

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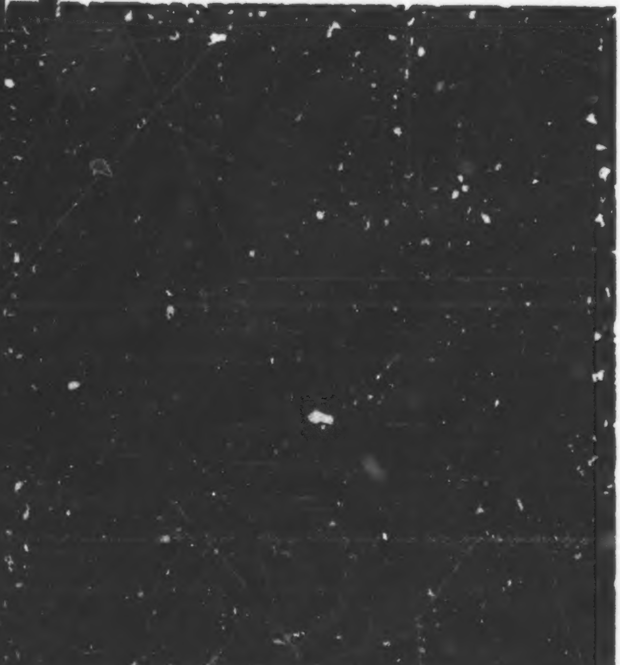

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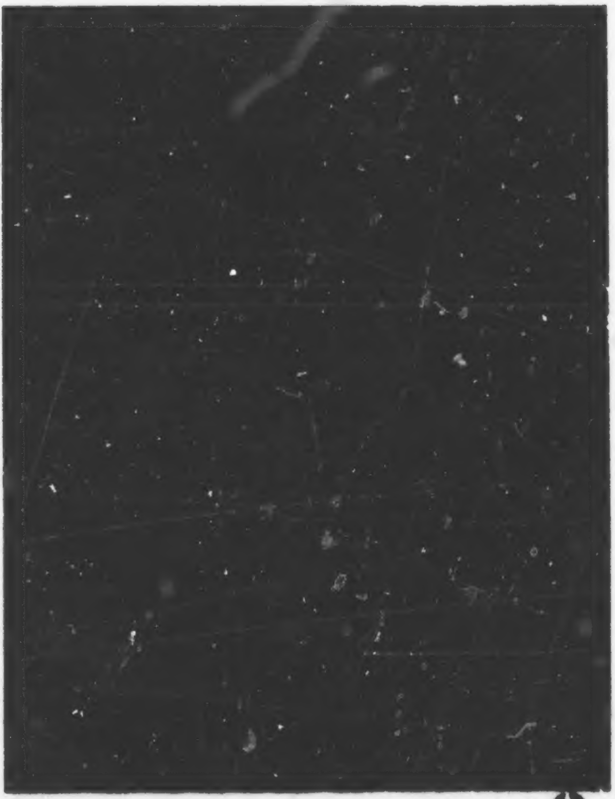

2

b. PHOTOMICROGRAPHS: Stone Dust

1. (Photo 0404-01) Particulate loading on Nuclepore filter: Stone dust used in CBDC mines (-200 mesh). x 200 (BEI). Note the bright grains indicating high Z elements.

2. Photo 0404-02) Enlargement of photo #1 showing range in particle size from approximately 1-20 microns. x 1000, (SEI)

c. EDX SPECTRA; Coal Minerals

1. Area raster on Low Temperature Ash (LTA) fragment of Photo #1. The ratios of peaks Si, Al, K, Fe, S - suggest the presence of mainly illite and pyrite. The overlapping gold peaks are an artifact of sputter coating.

2. Spot analysis on an illite flake (photo #2). This is a typical profile for the illite clay member, except for the gold peaks.

3. Spot analysis on a framboid crystal. The gold and sulphur peaks overlap, otherwise it is a typical pyrite spectrum. Note, however, that the crystal face is perpendicular to our beam (cross hairs). See photo #8.

4. Spot analysis on massive pyrite. Some clay contamination is evidenced by the Al and Si peaks. Photo #5.

5. Spot analysis on "pyrite" framboids. The absence of a sulphur peak indicates this is an LTA oxidation product pseudomorphic after pyrite. The presence of a strong Si peak and weak Cu peak illustrates the problem of artifacts when analysing particulates of uncontrolled geometry. The Si and Ci peaks are due to stray electrons hitting the BEI detector.

6. Spot analysis repeat of EDX spectrum #5 but with the BEI detector removed. The Cu and Si peaks are much reduced.

7. Spot analysis on crystals of a pyrite framboid. Different crystals show different Fe:S ratios, either due to geometry effects (ie. x-ray absorption) or due to degrees of oxidation (photo #7).

8. Same as above.

9. Same as above.

10. Raster analysis on the same framboid as for EDX 7 to 9. An "average" Fe:S ratio is obtained. But are we averaging the chemistry or the absorption effects?

11. Typical area raster spectrum for a polished pellet of bituminous coal. (Minto-Chipman #1). Carbon coated. Note the large background due to the presence of amorphous organic material. The strong sulphur peak is due to both organic S and pyrite. (photo #9).

12. Typical spectrum for a coal maceral (photo #11). The high S and Si is dominantly in the organic matrix although some submicron size mineral grains may be included.

13. EDX spectrum for illite flake (grain 2, photo #11). The ratios of Al/K and Al/Si are diagnostic. Note the "clean" spectrum obtained from a polished pellet coated with carbon.

14. EDX spectrum of quartz intergrown with illite and traces of pyrite (grain 3, photo #11).

15. EDX spectrum of massive pyrite (photo #12). The spot analysed was near the inclusion (above the cross hairs) of pentlandite (?) as shown by the Ni peaks.

16. EDX spectrum of oxidation product on pyrite LTA: likely hematite. The presence of Cu and Si is due to instrument artifacts.

17. EDX spectrum of zircon (grain 1, photo #18) from polished pellet of Evans #5 coal, carbon coated. Note the clean spectrum with essentially no artifacts and low background. Such mineral grains account for most of the trace elements reported for coal.

8-27. EDX spot analyses on various crystal faces of pyrite (photo#14). The orientation of the detector in relation to the beam spot and crystal face dictates the ratio of Fe:S. The x-ray detector is positioned at the top right corner of the photo (opposite side to micron marker). The most accurate analyses are represented by tests #1 and #8 with crystal faces perpendicular to beam. Tests #2 and #9 represent extreme conditions of sulphur x-ray absorption because the detector is out of view of the crystal face. These tests illustrate the difficulties of differentiating between pyrite, pyrrhotite and troilite or other chemically similar minerals when geometry cannot be controlled. Such effects must be considered for respirable dust analysis.

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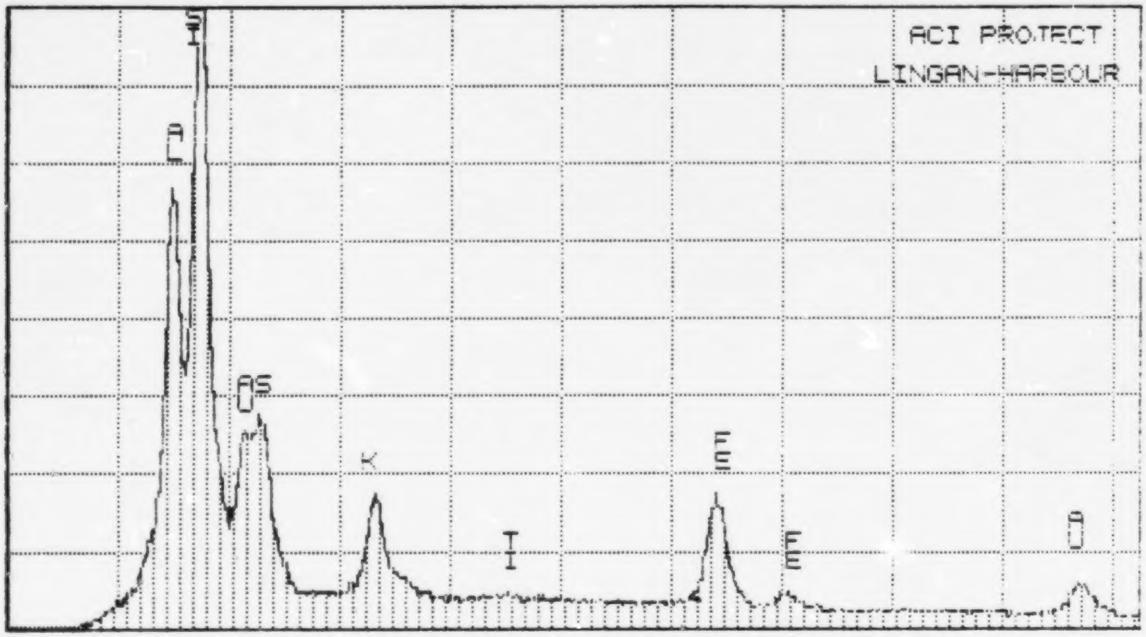

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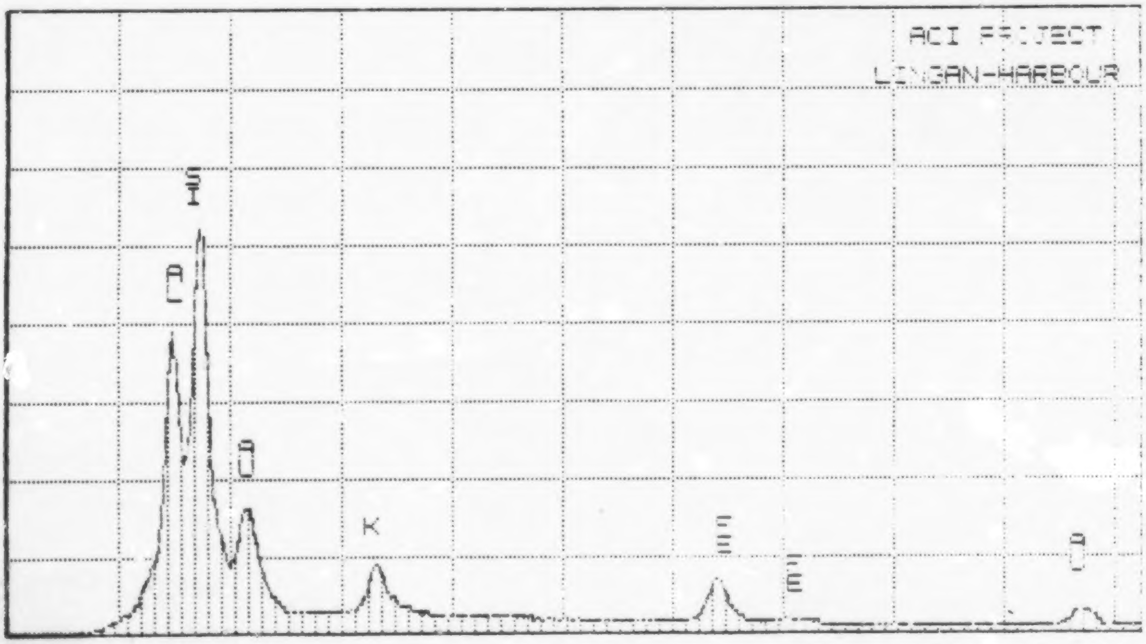

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2

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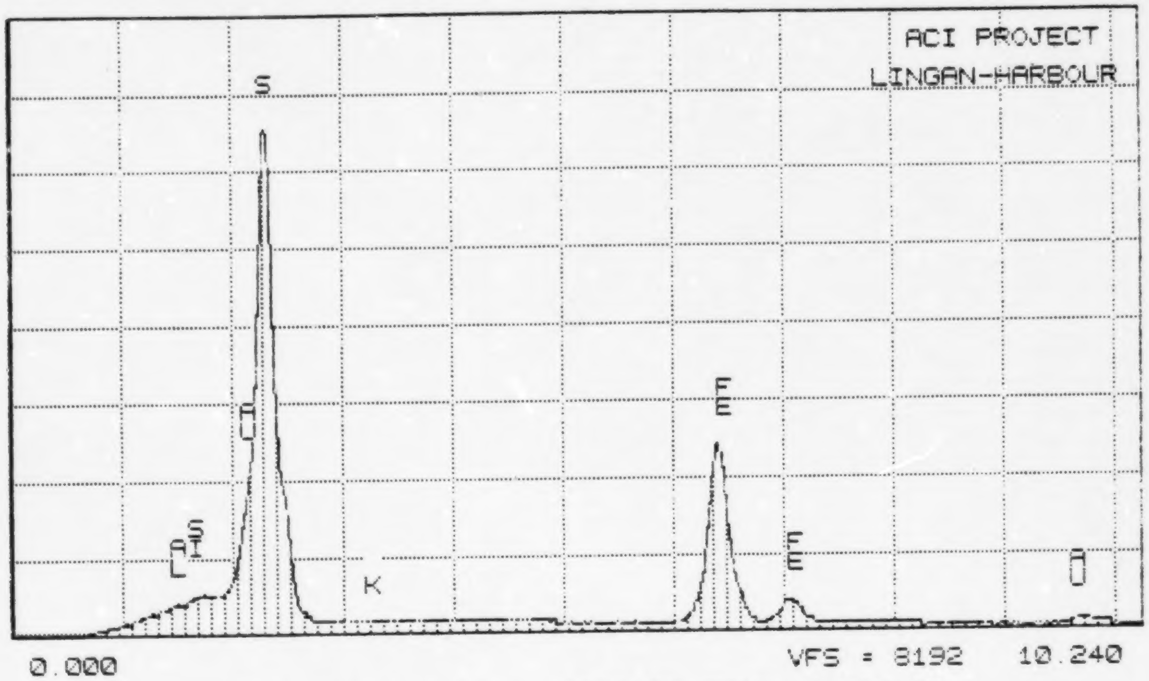

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3

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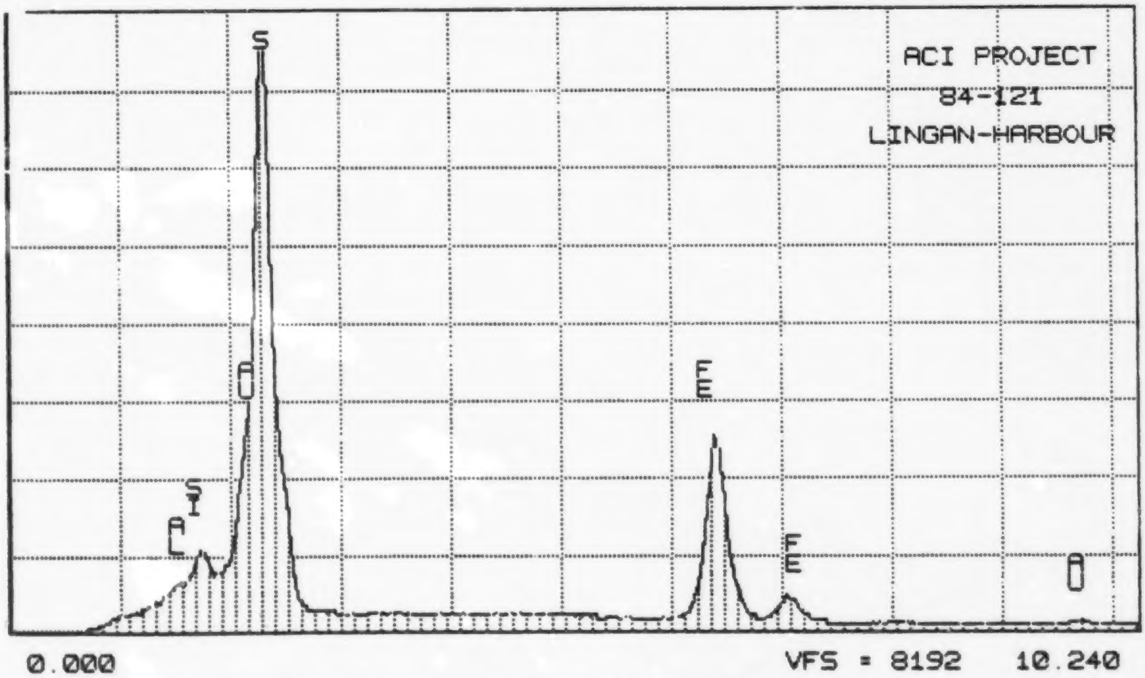

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4

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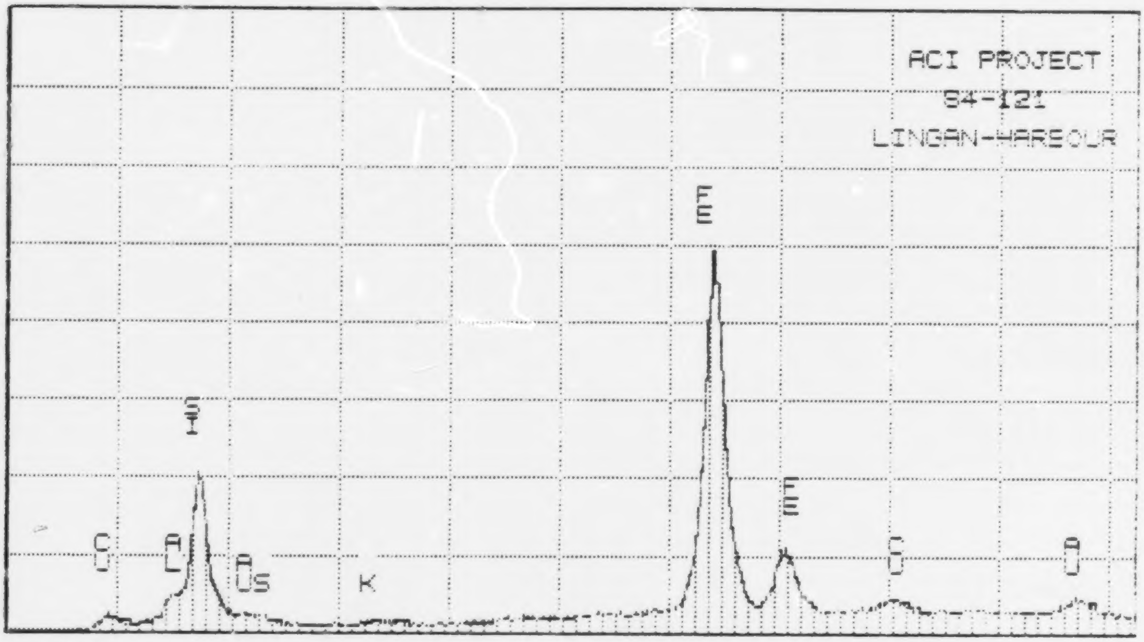

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5

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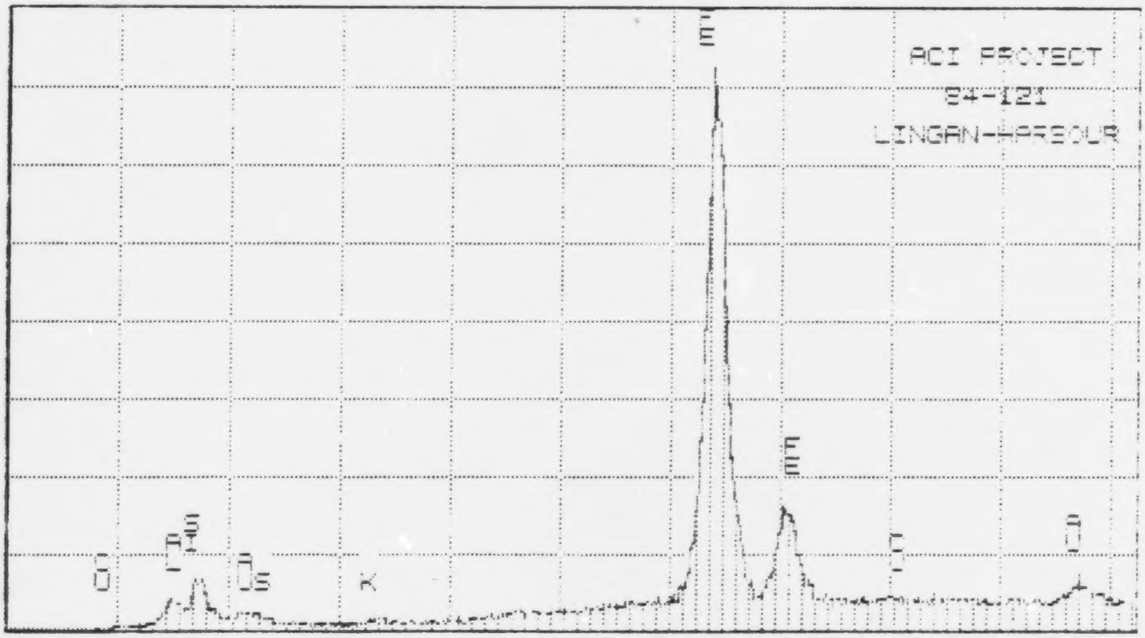

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6

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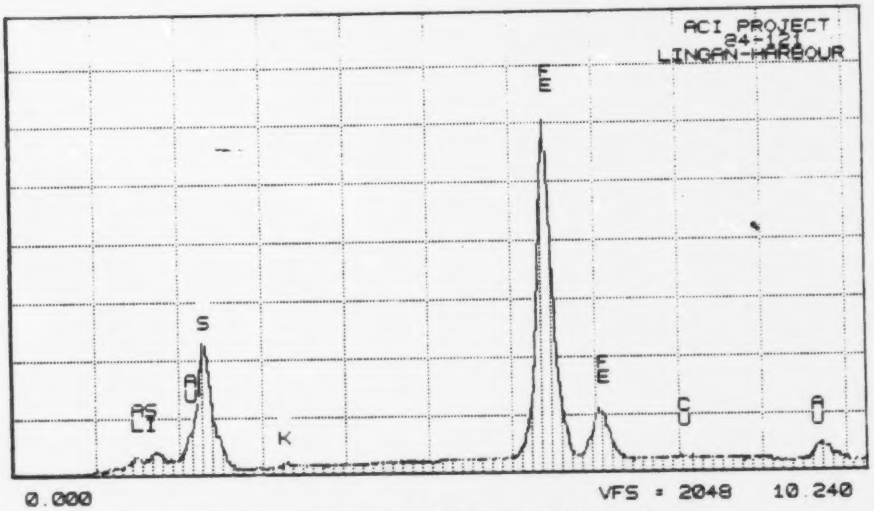

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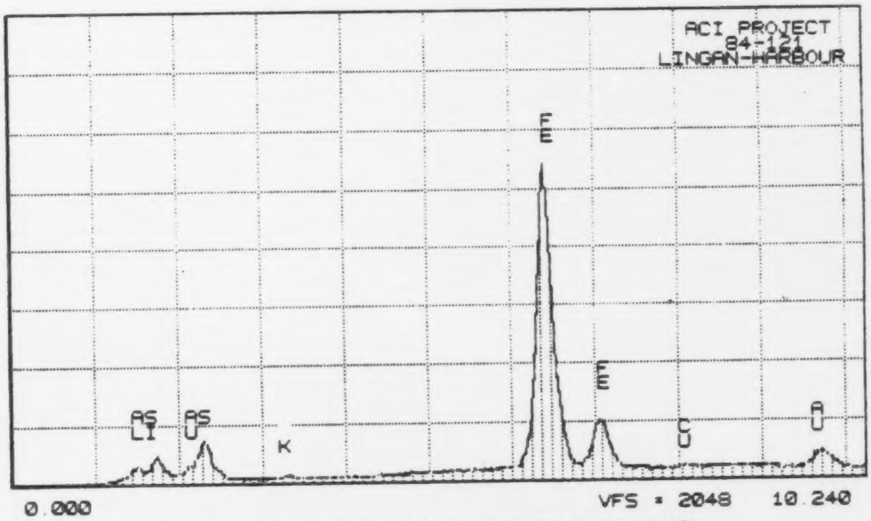

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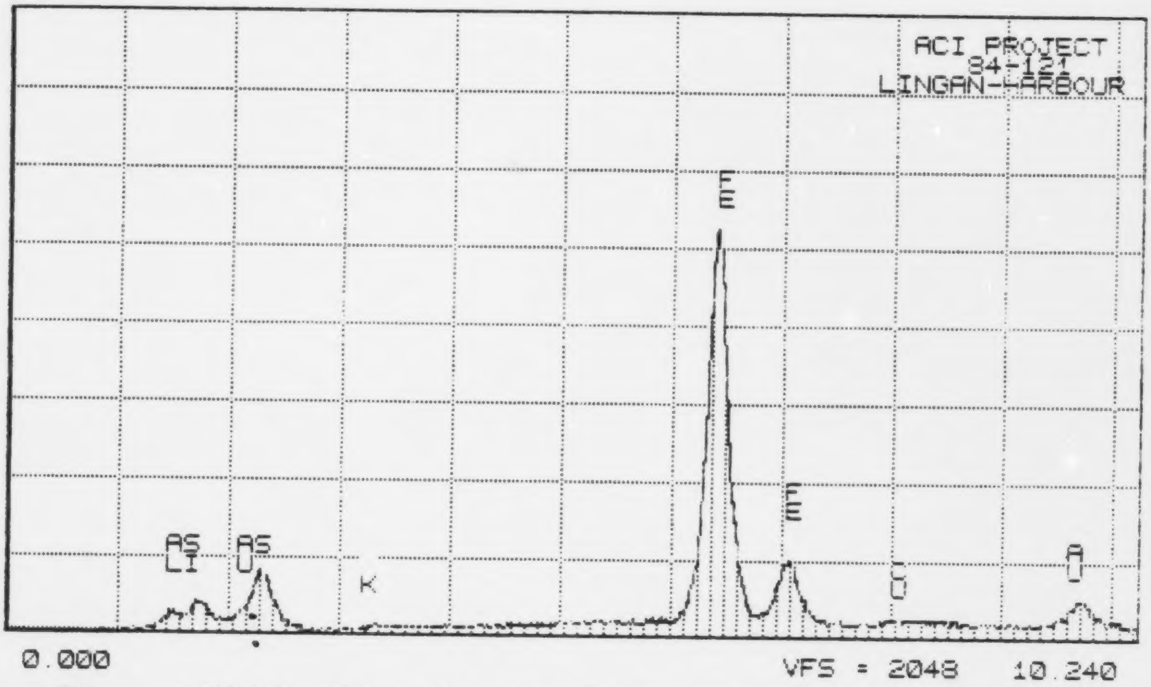

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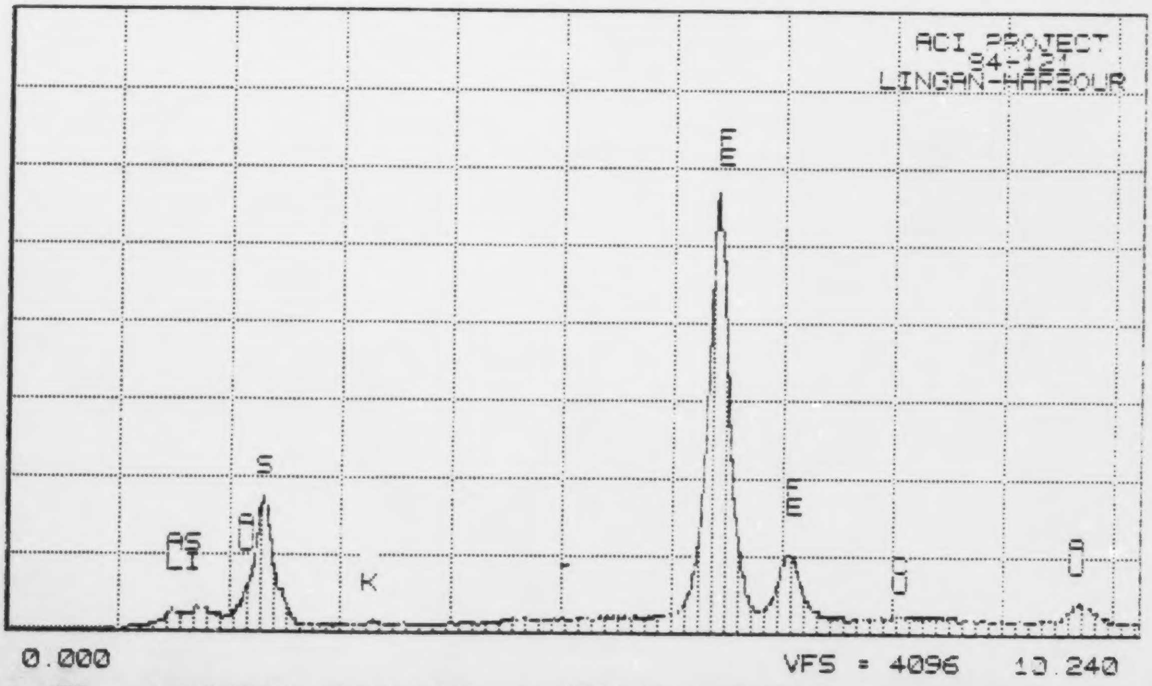

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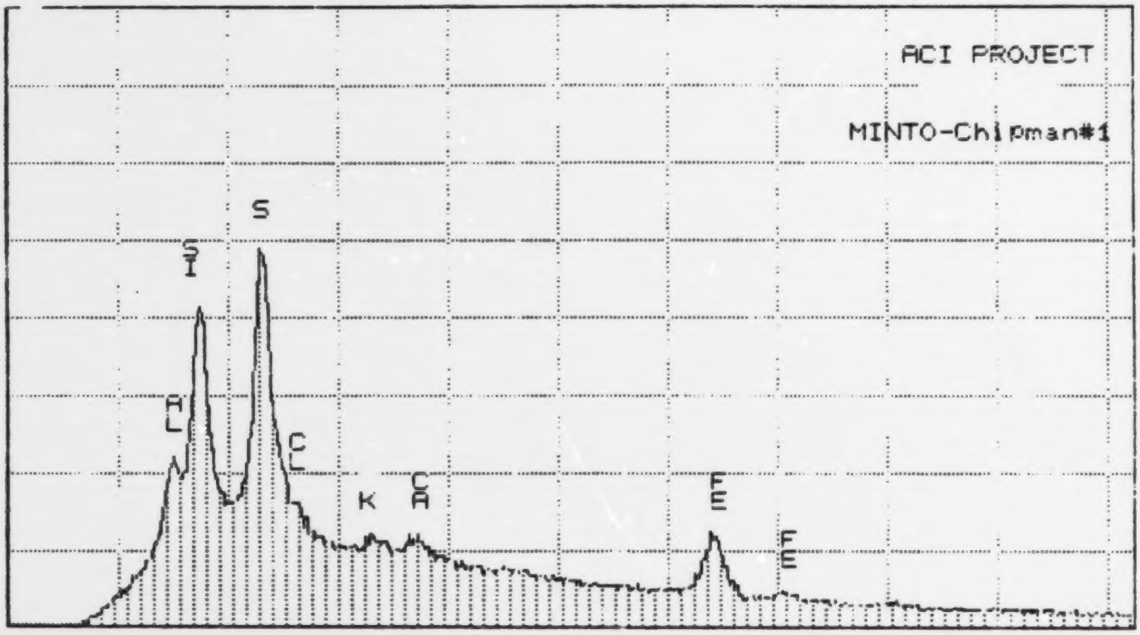

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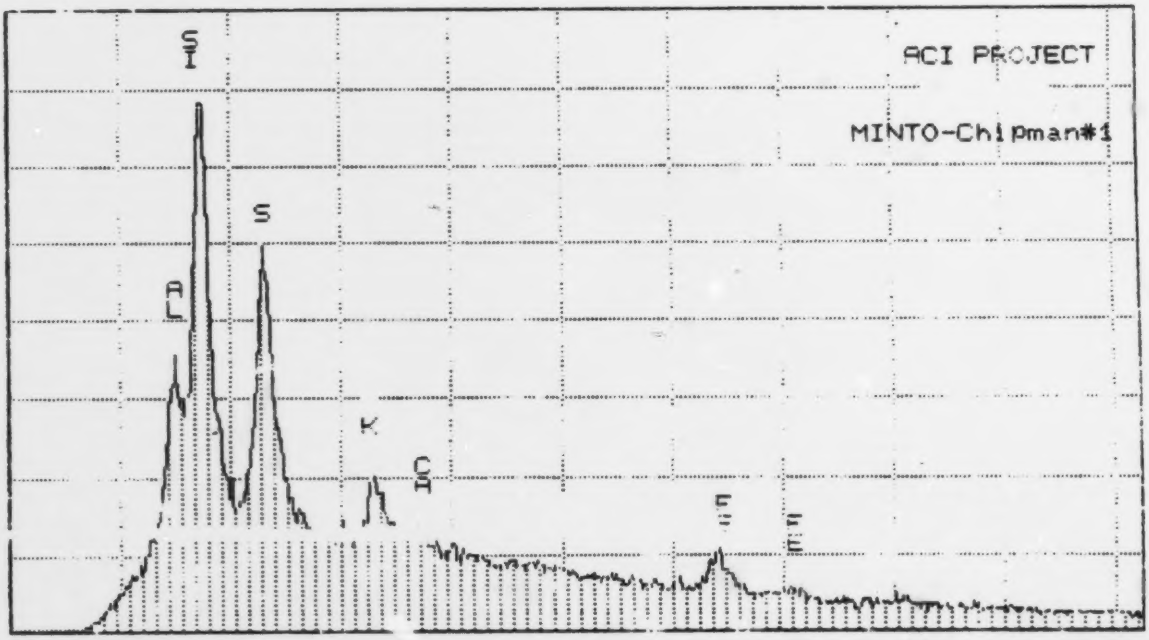

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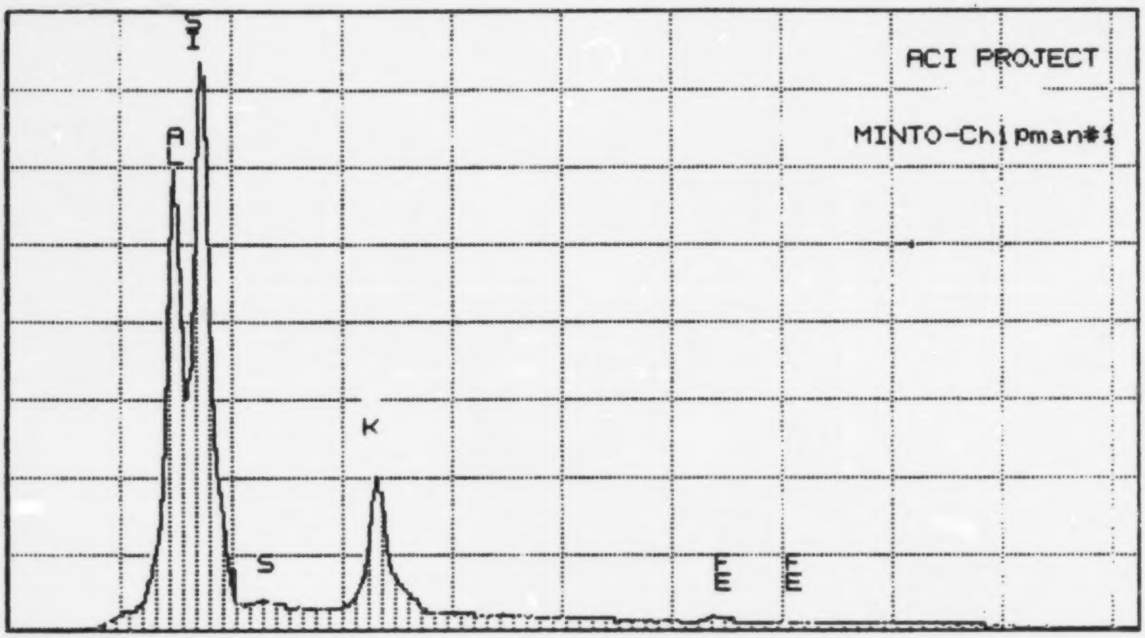

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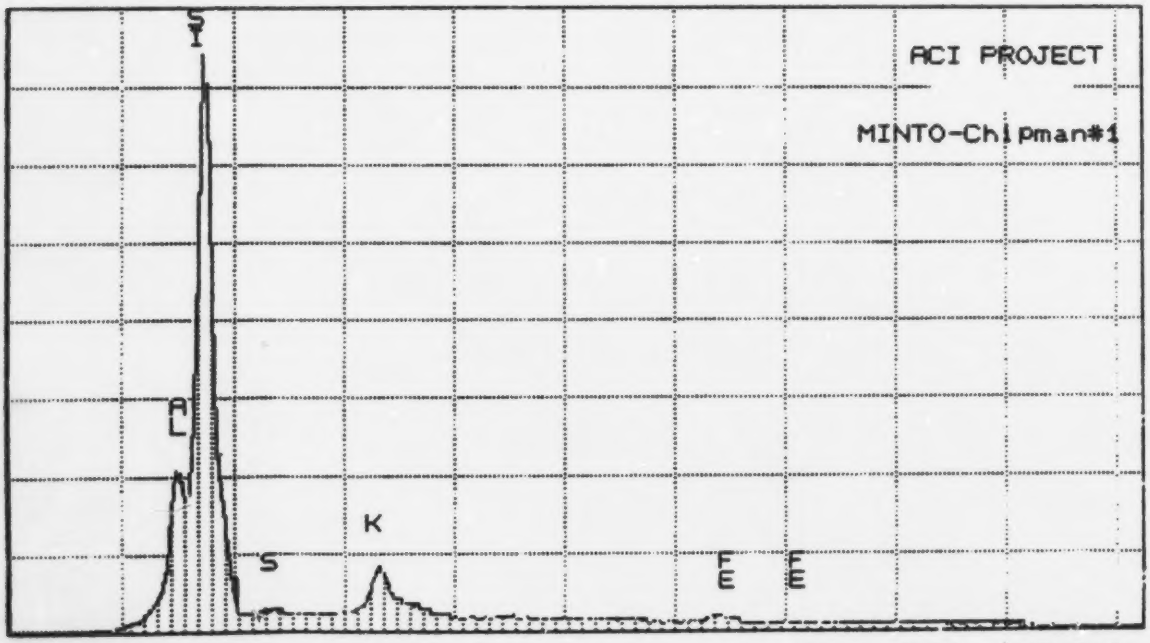

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14

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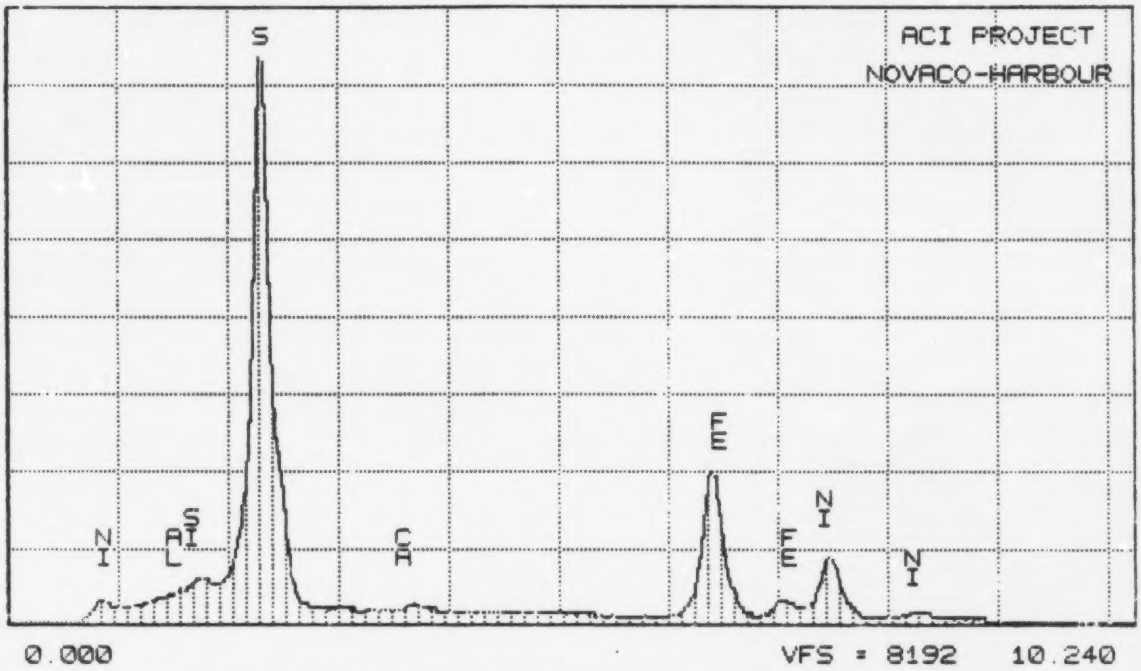

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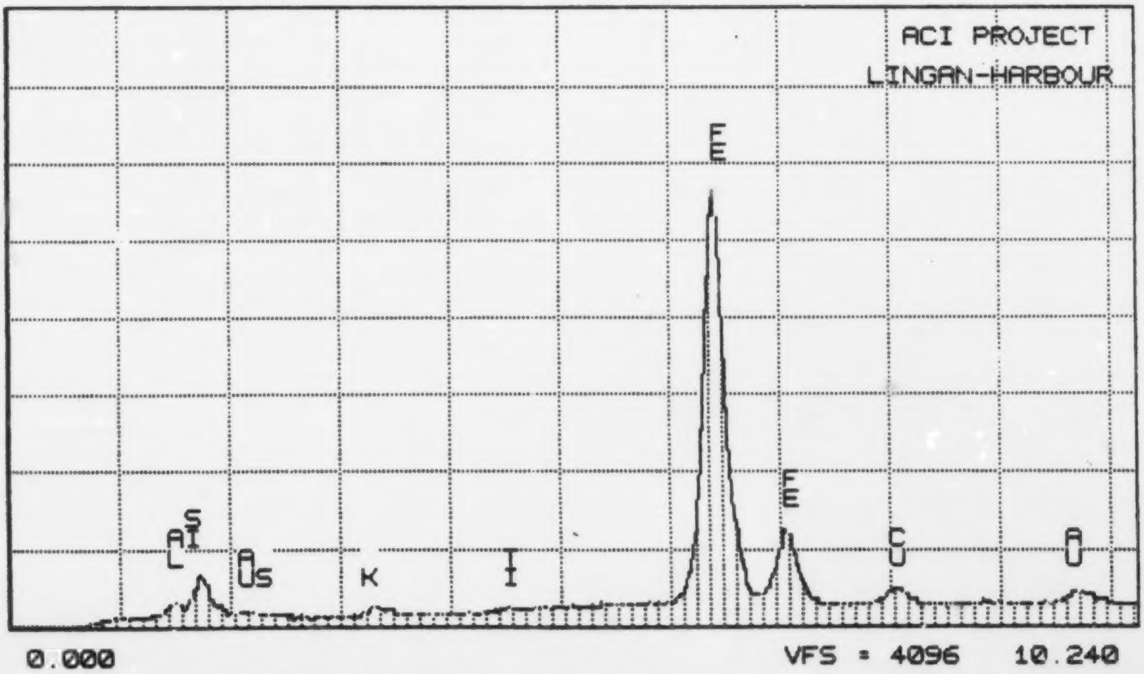

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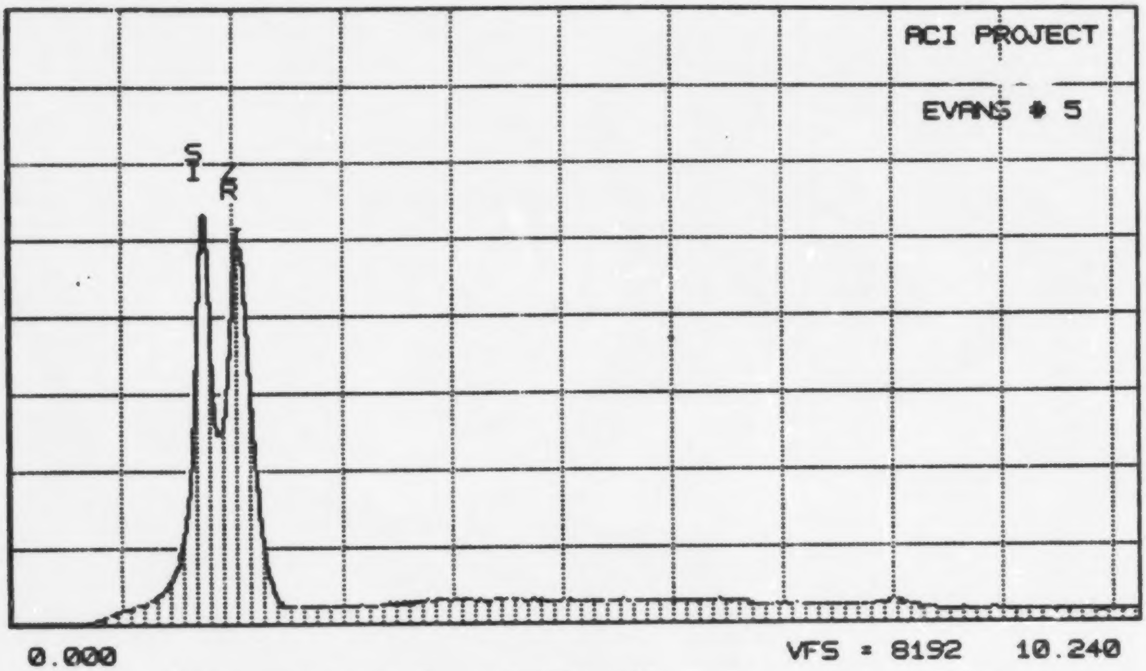

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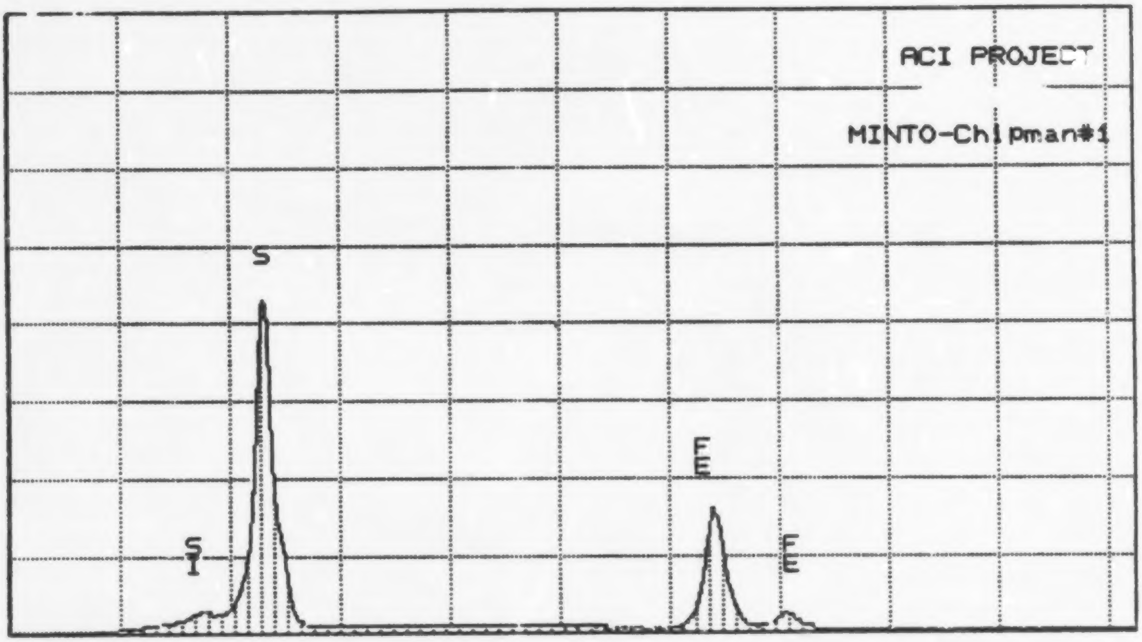

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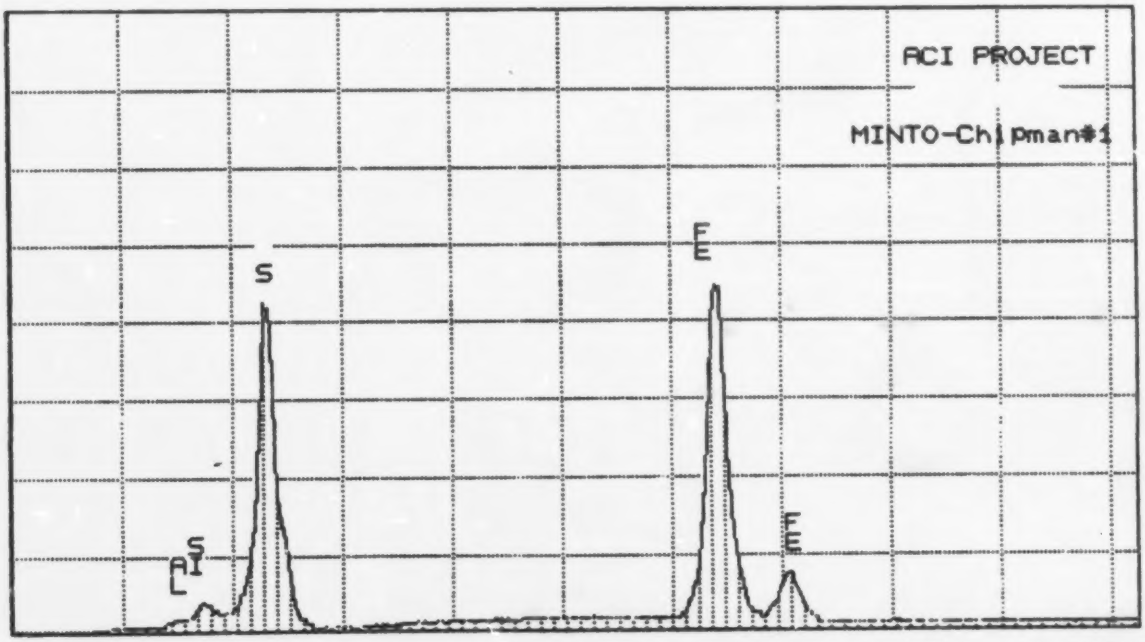

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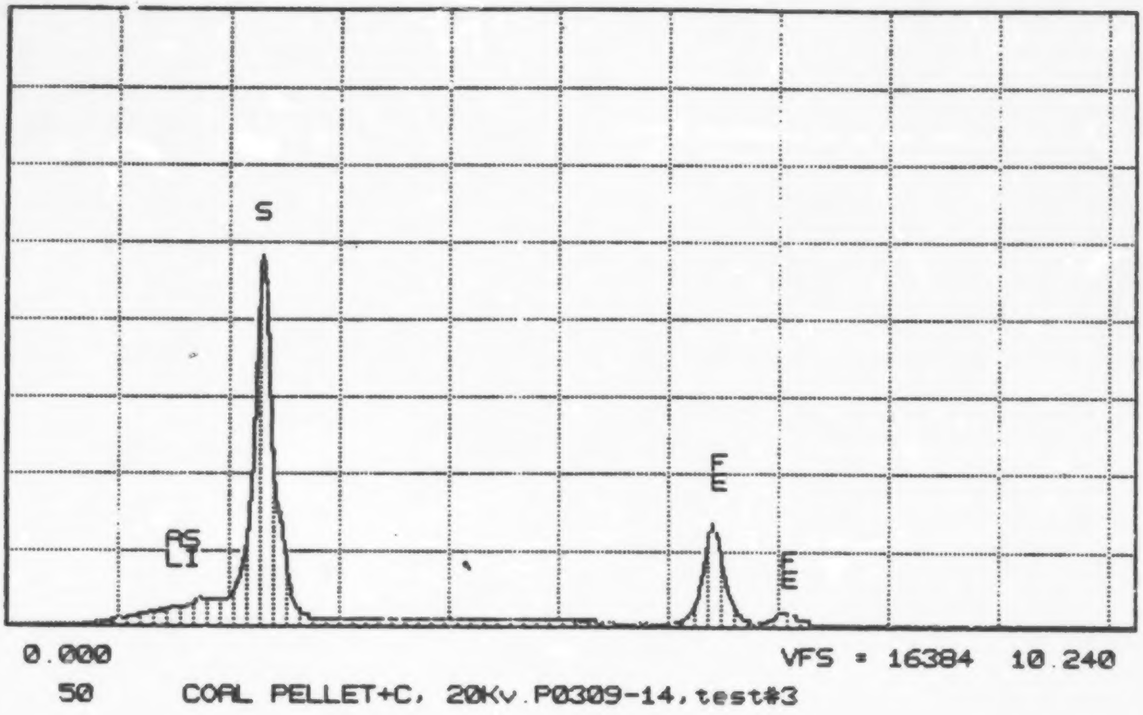

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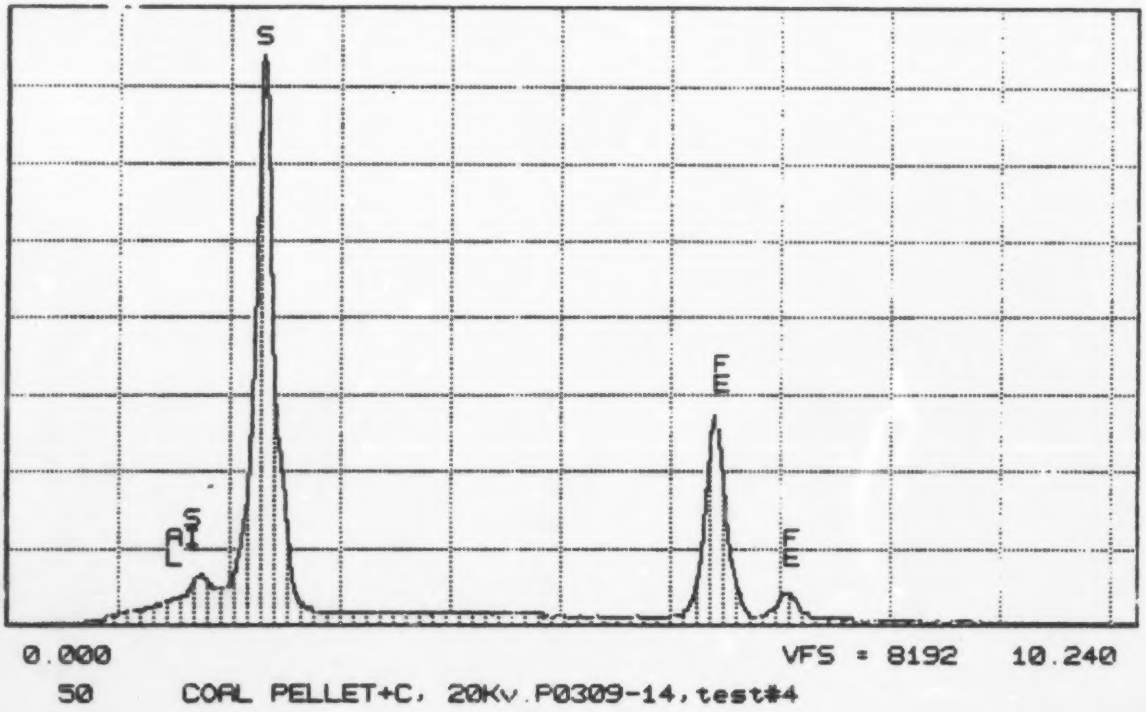

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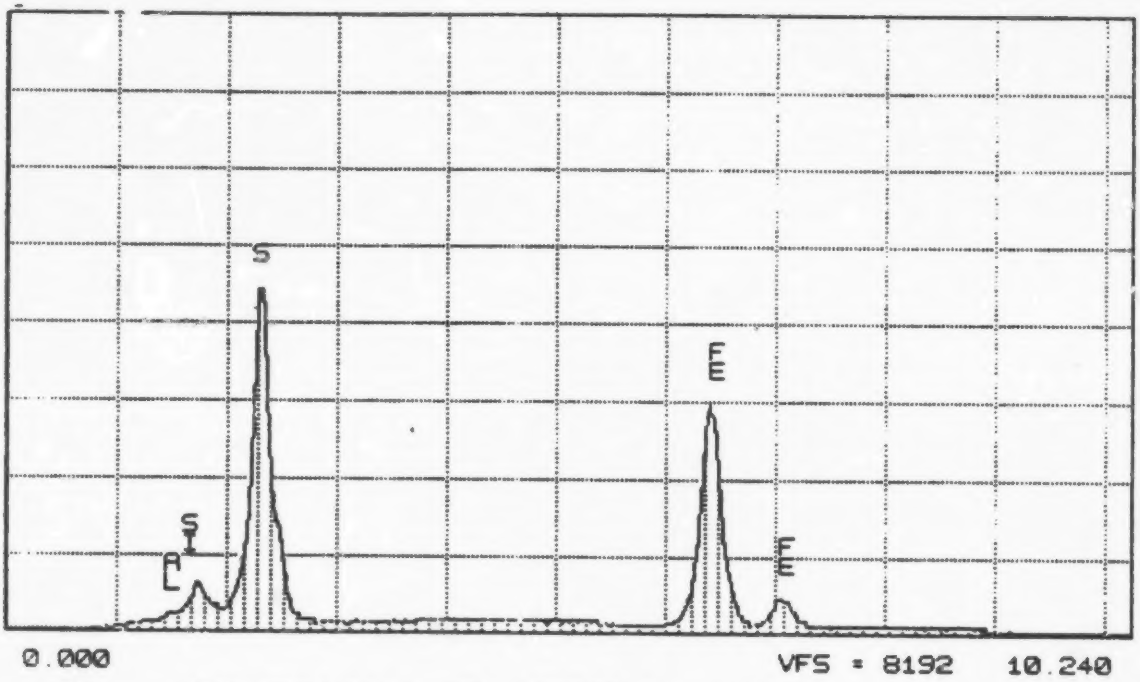

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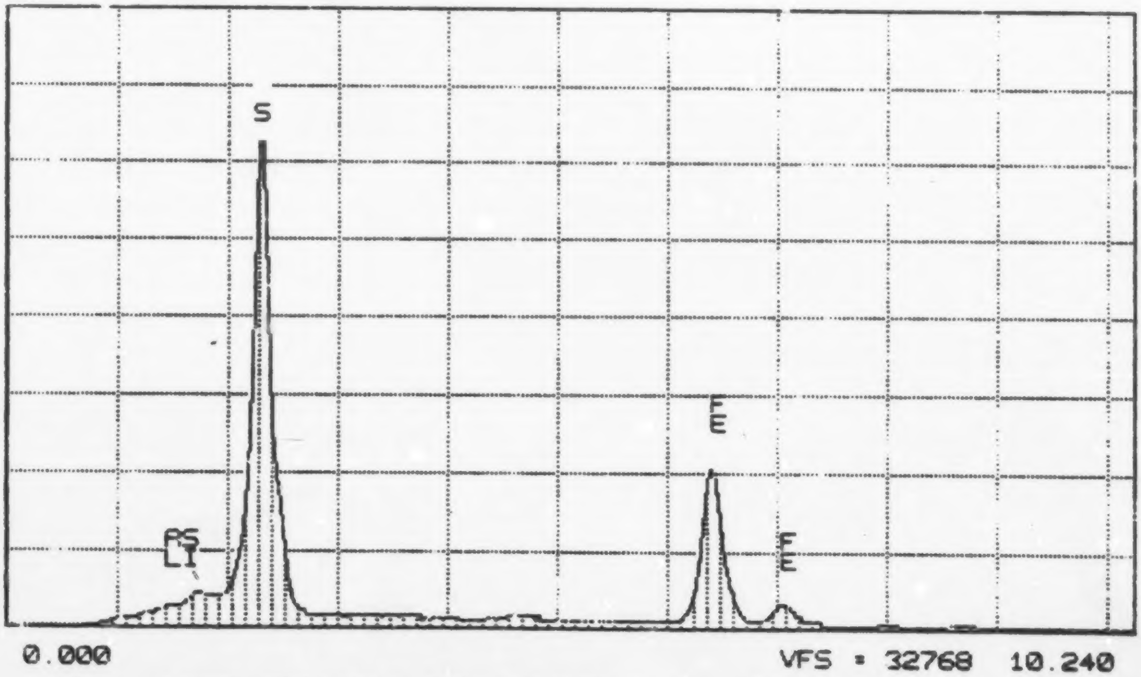

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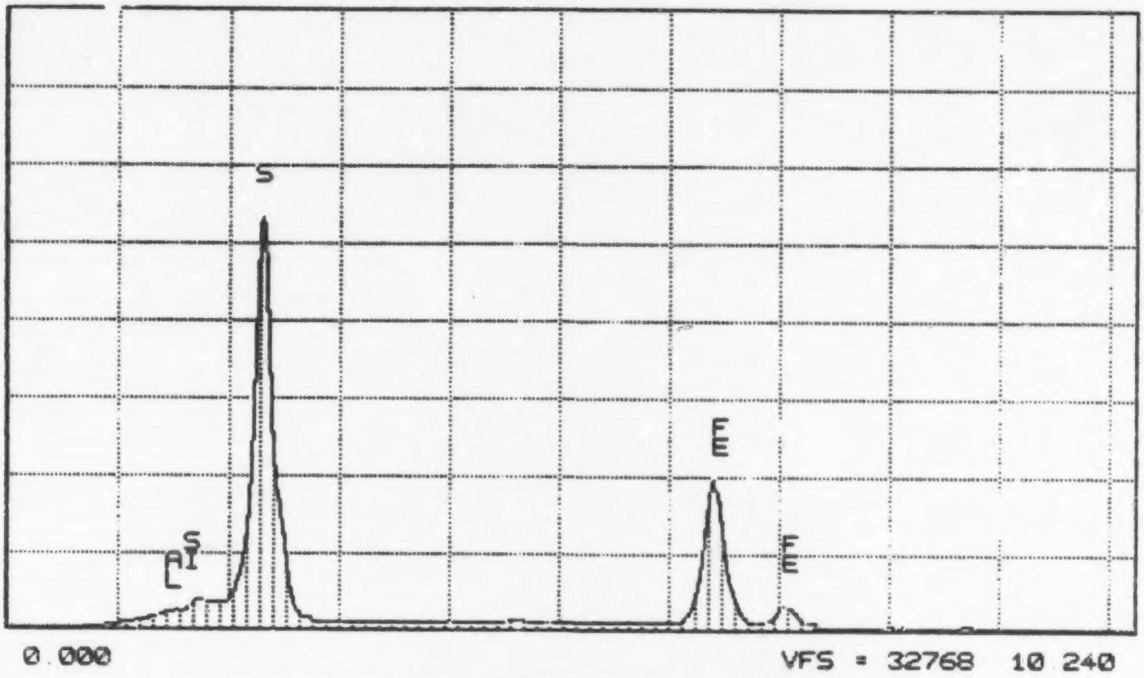

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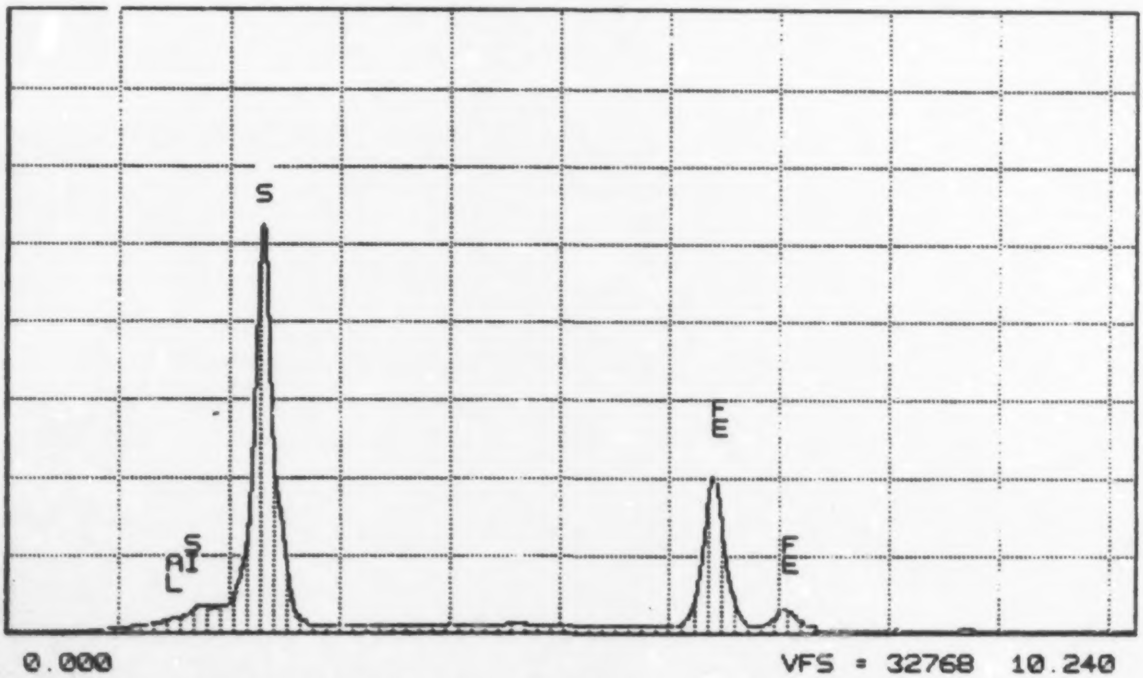

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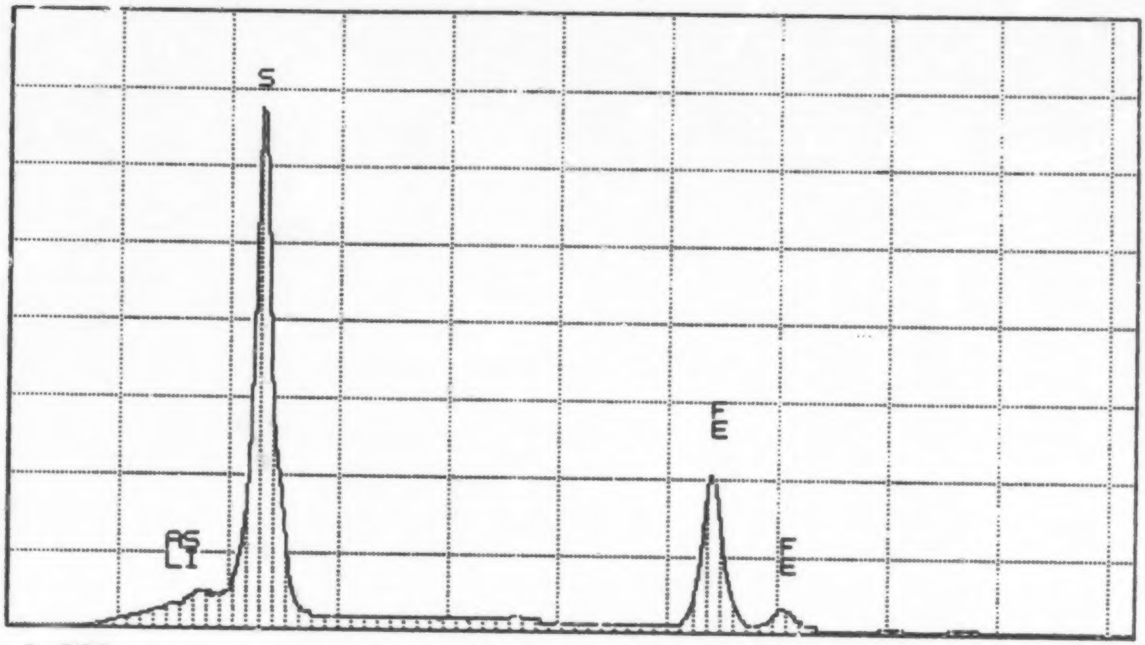

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26

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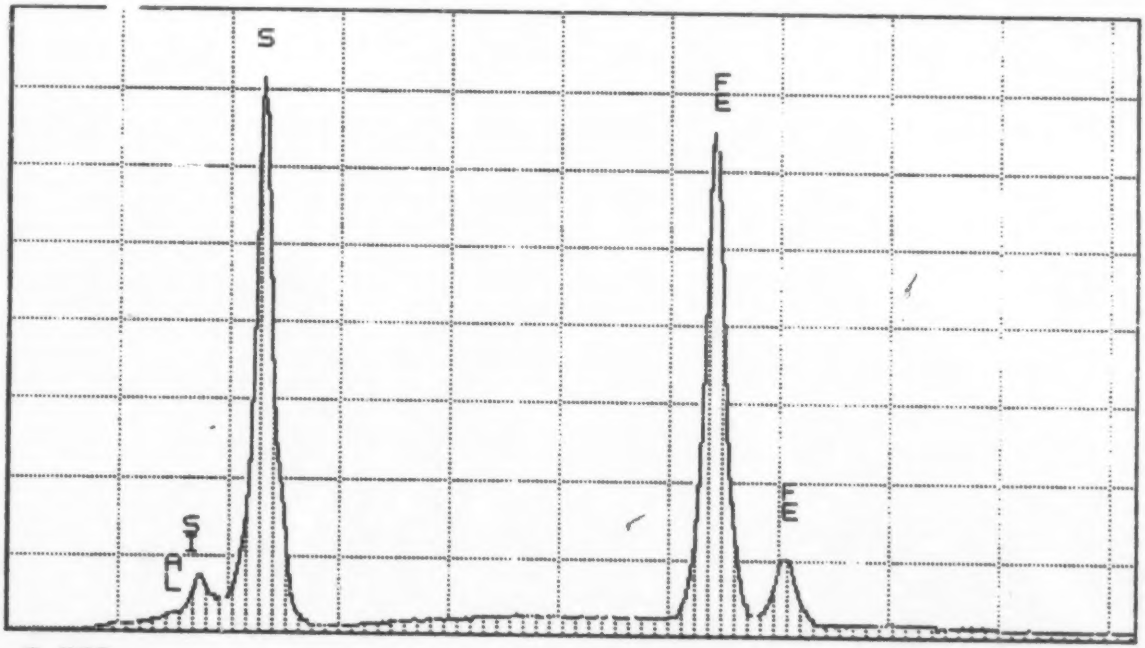

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27

d. EDX SPECTRA: Stone Dust

1. EDX spectrum from spot analysis of stone dust particle (photos 1,2) at 20kv for 10 second counts. The majority of particles show only a Ca peak indicative of calcite or aragonite ($CaCO_3$). Since aragonite is unstable and converts to calcite in contact with water, we assume all such spectra are calcite.
2. EDX spectrum (spot analysis) of a bright grain (BEI mode) showing large Fe contribution, likely siderite. The abundance of other elements is either spurious due to particulate geometry or indicates a mineral mixture.
3. EDX spectrum of a barium mineral, likely barite, but with a large Ca contribution. The presence of Ca in all the spectra for stone dust particles is likely due to finer calcite dust on these minerals or due to the rough geometry and spurious x-rays from adjacent grains. Mineral identification is therefore tentative as indicated by (?). When isolated in a coal dust matrix such grain spectra would be more diagnostic.
4. Ilmenite (?) $FeTiO_3$
5. Collophane (?) Ca (OH, F, C) (PO)
6. Celestite (?) $SrSO_4$
7. Siderite (?) $FeCO_3$
8. Siderite (?)
9. Pyrite (?)
10. Sphalerite (?)
11. Zircon (?) $Zr SiO_4$

ATLANTIC COAL INSTITUTE MON 08-APR-85 12:19

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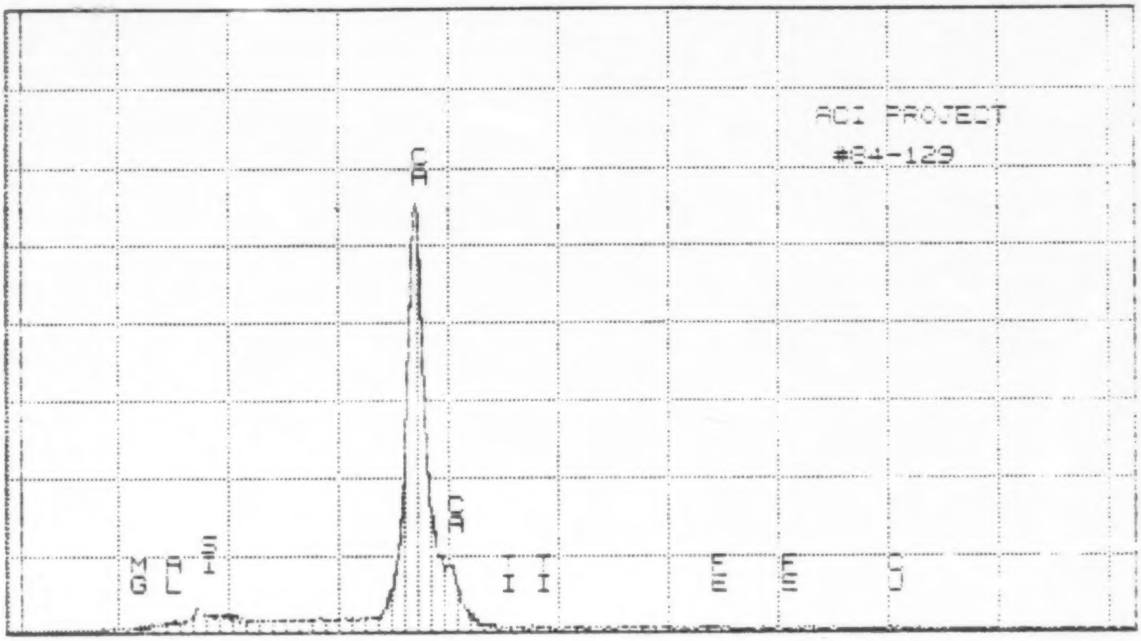

0.000 VFS = 2048 10.240

10 DUST STONE DUST SAMPLE FROM CBCAL

1

ATLANTIC COAL INSTITUTE THU 04-APR-85 16:13

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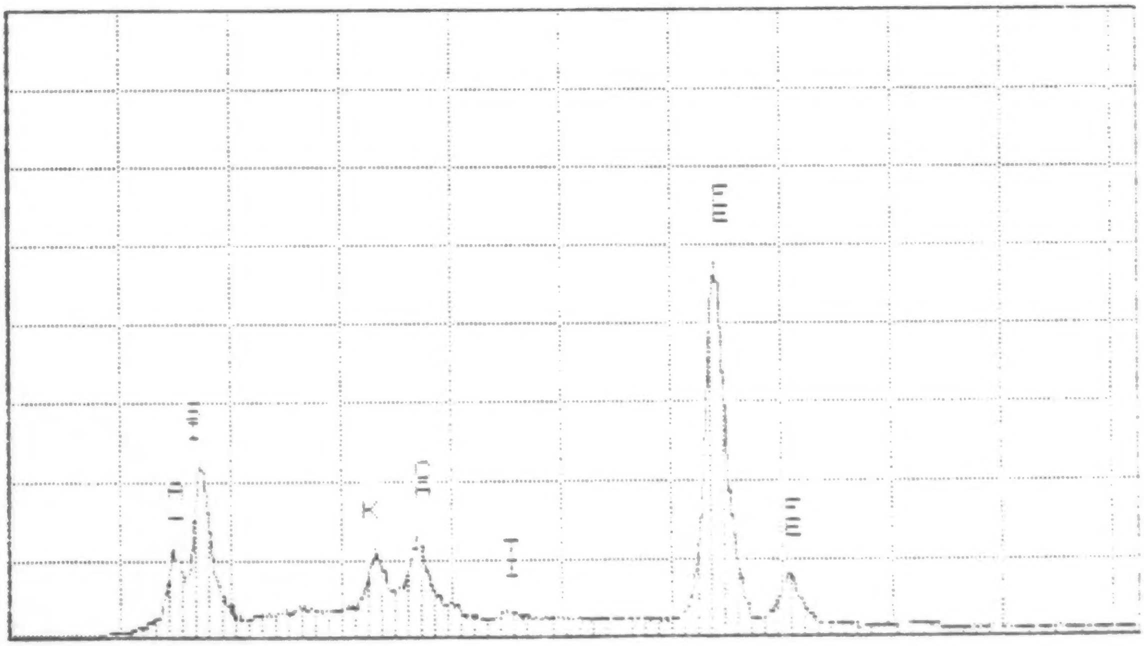

0.000 VFS = 2048 10.240

10 DUST STONE DUST SAMPLE

2

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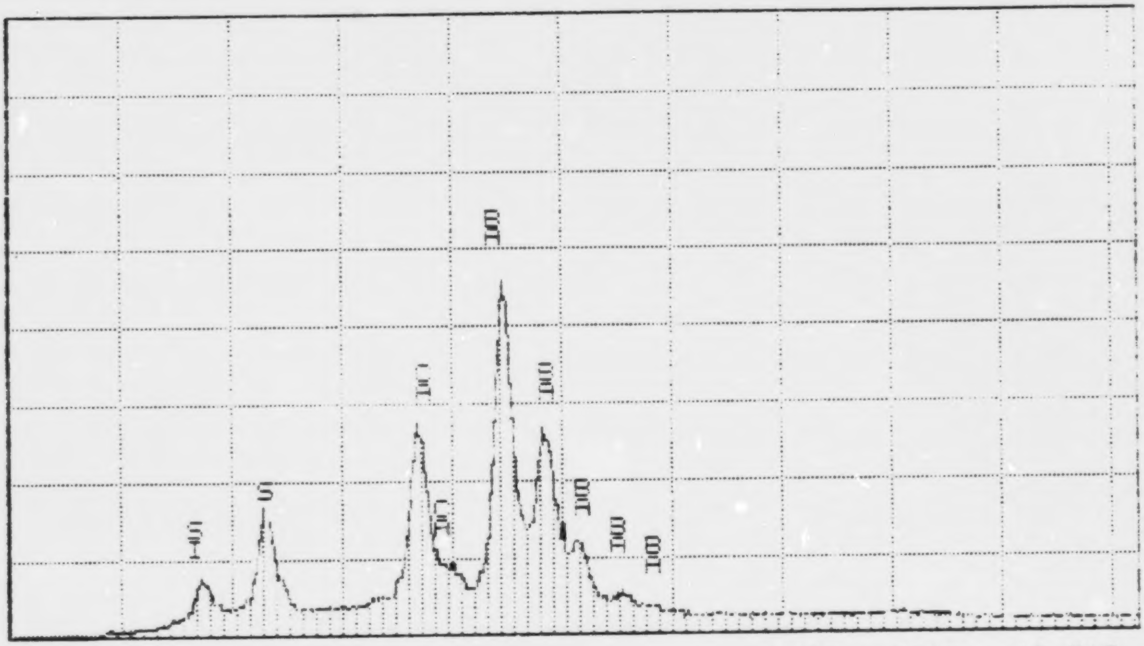

0.000 VFS = 2048 10.240

10 STONE DUST SAMPLE FROM CBCRL

3

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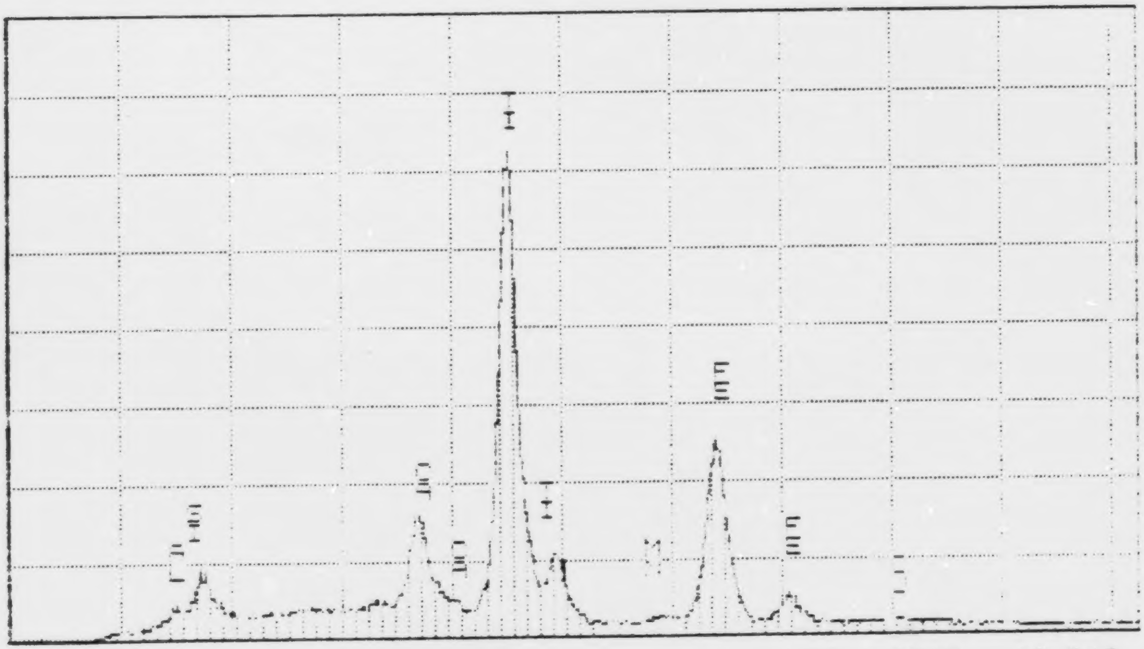

0.000 VFS = 2048 10.240

10 STONE DUST SAMPLE FROM CBCRL

4

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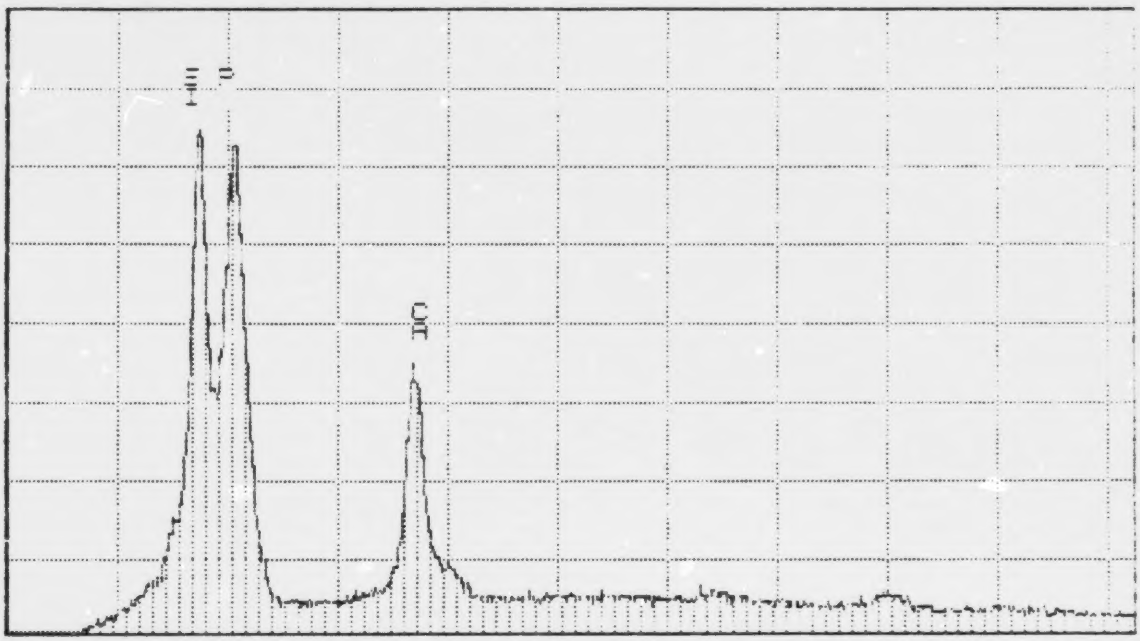

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   10          STONE DUST
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5

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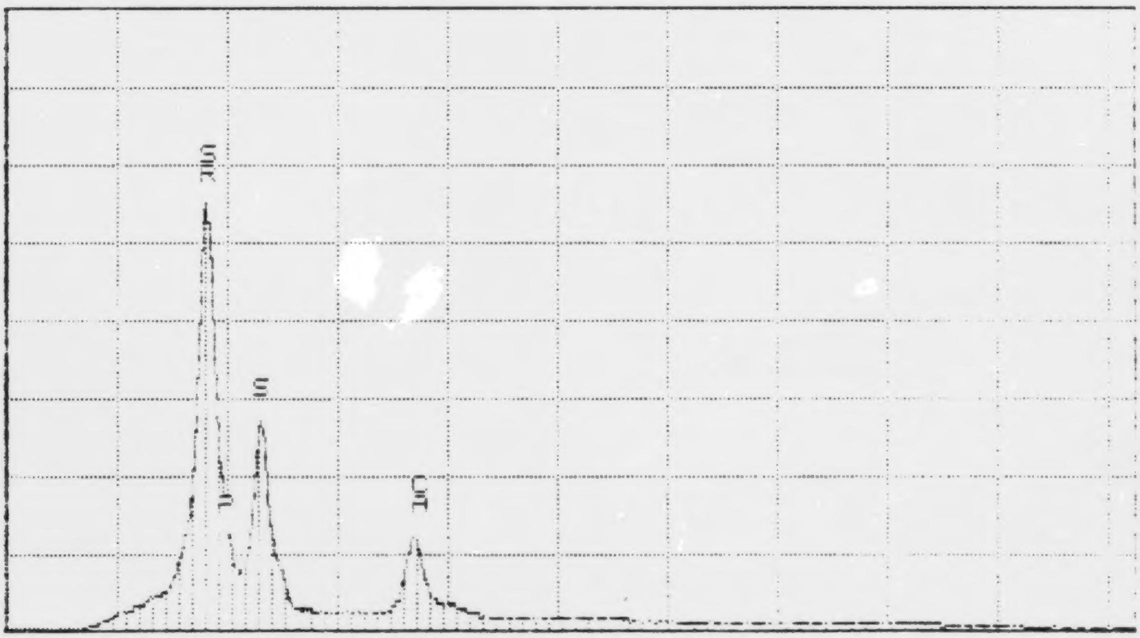

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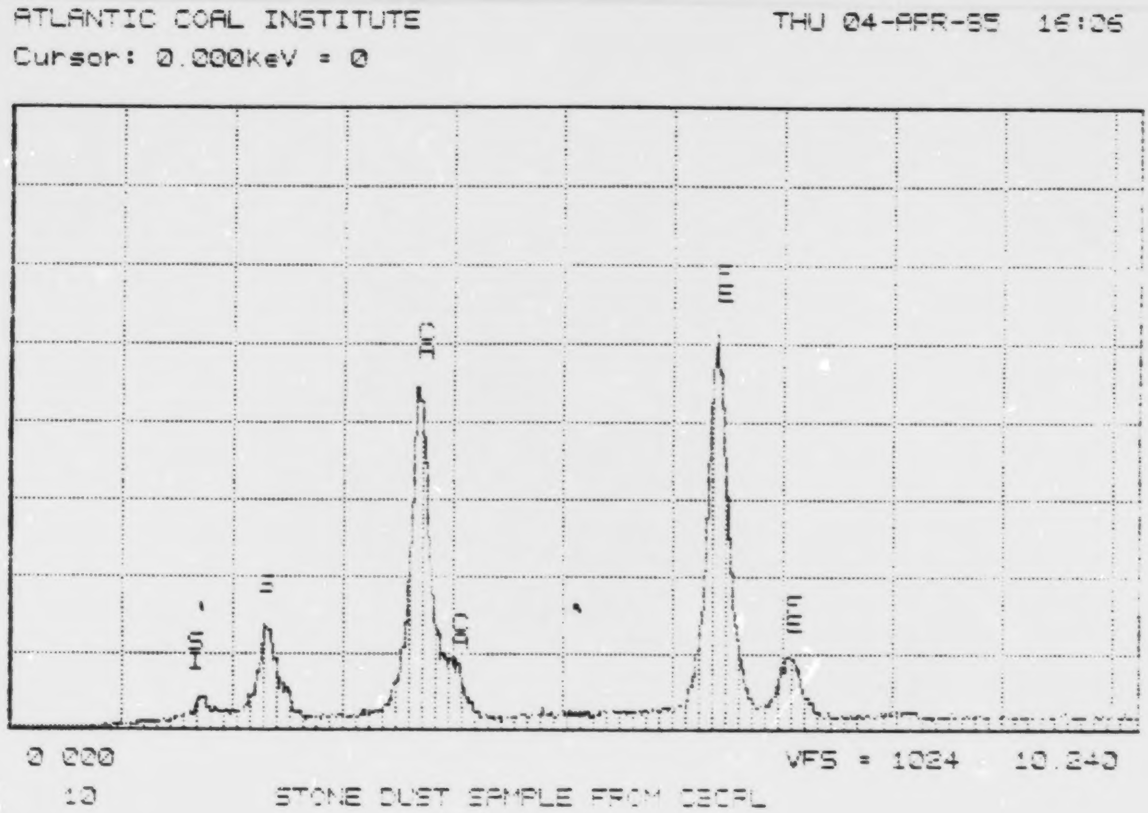
   10          STONE DUST
```

6



ATLANTIC COAL INSTITUTE THU 04-APR-85 16:06

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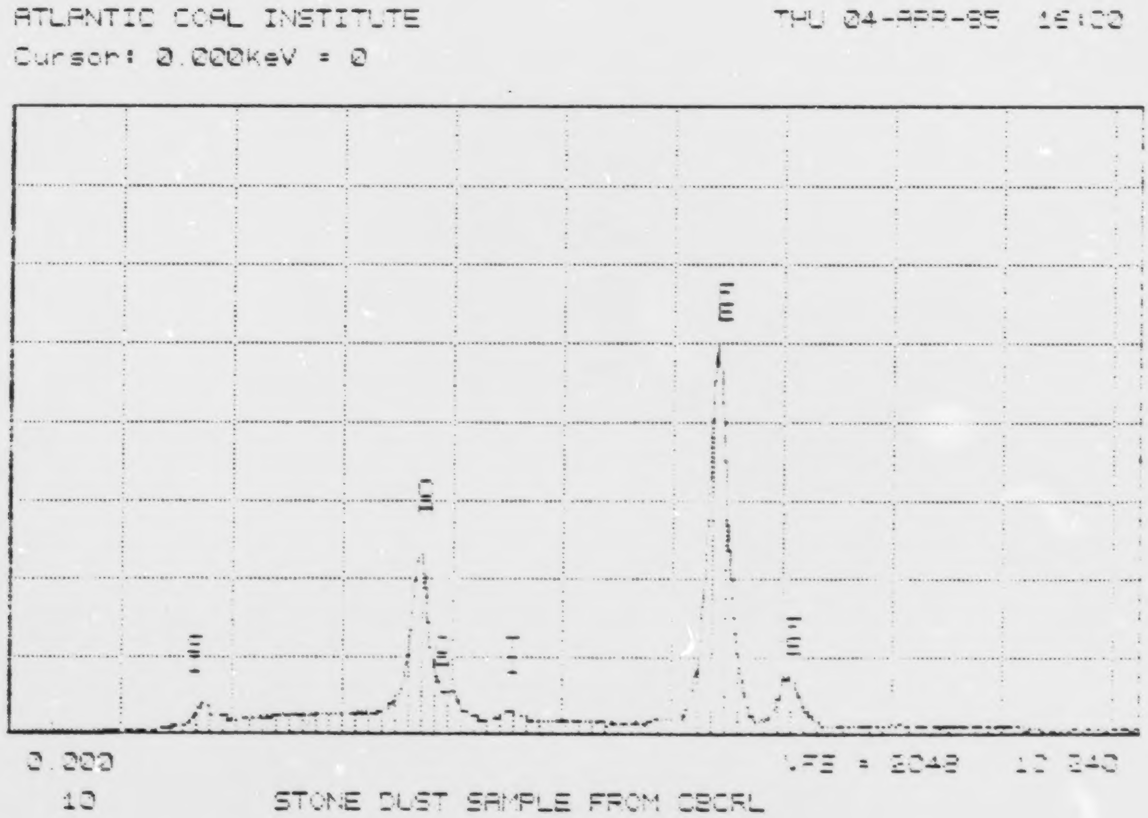
0.000 VFS = 1024 10.240

10 STONE DUST SAMPLE FROM CBCRL

7



ATLANTIC COAL INSTITUTE THU 04-APR-85 16:00

Cursor: 0.000keV = 0

0.000 VFS = 2048 10.240

10 STONE DUST SAMPLE FROM CBCRL

8

ATLANTIC COAL INSTITUTE MON 08-APR-85 11:55
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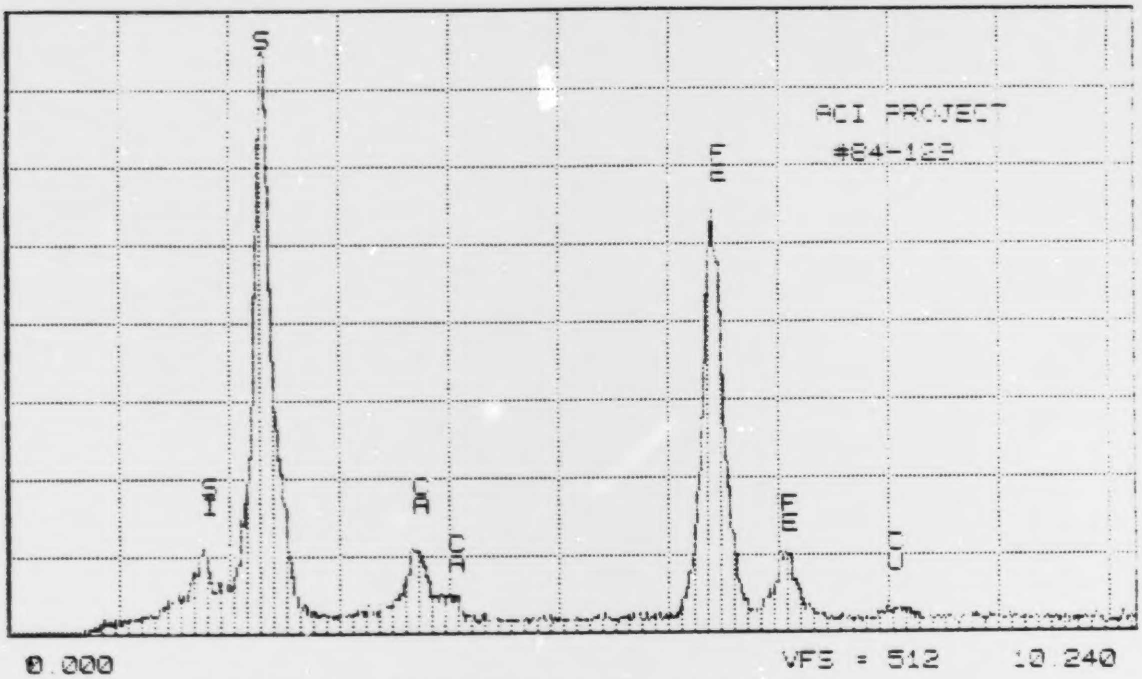

10 DUST STONE DUST SAMPLE FROM CSCRL

9

ATLANTIC COAL INSTITUTE MON 08-APR-85 12:11
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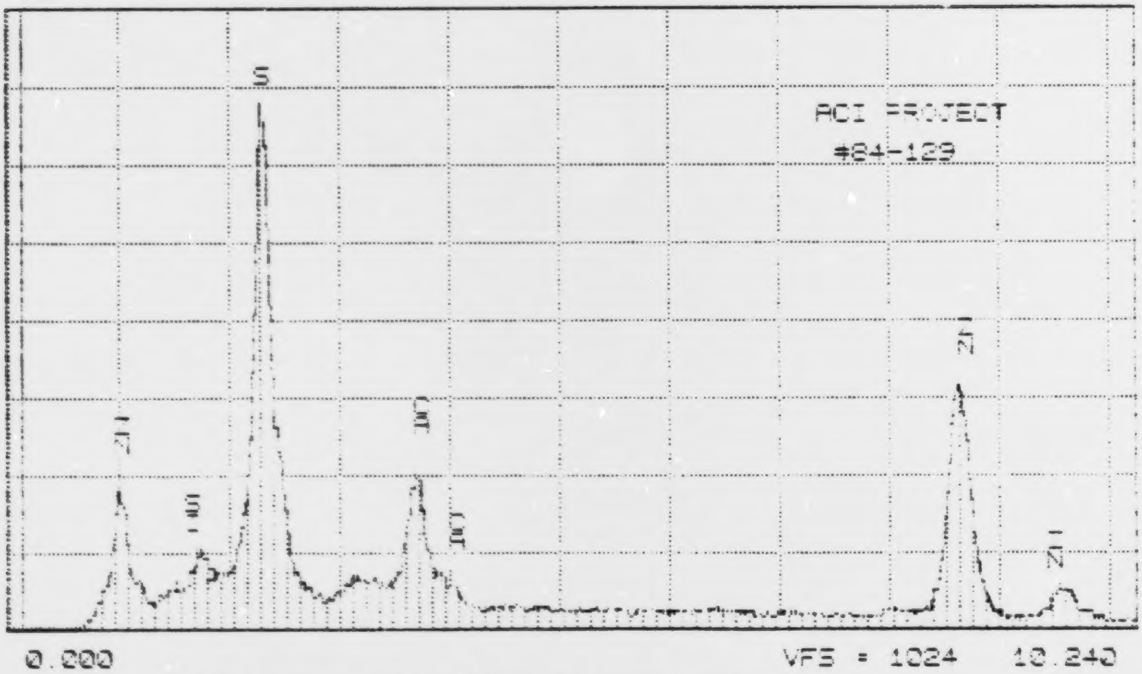

10 DUST STONE DUST SAMPLE FROM CSCRL

10

ATLANTIC COAL INSTITUTE MON 08-APR-85 13:57

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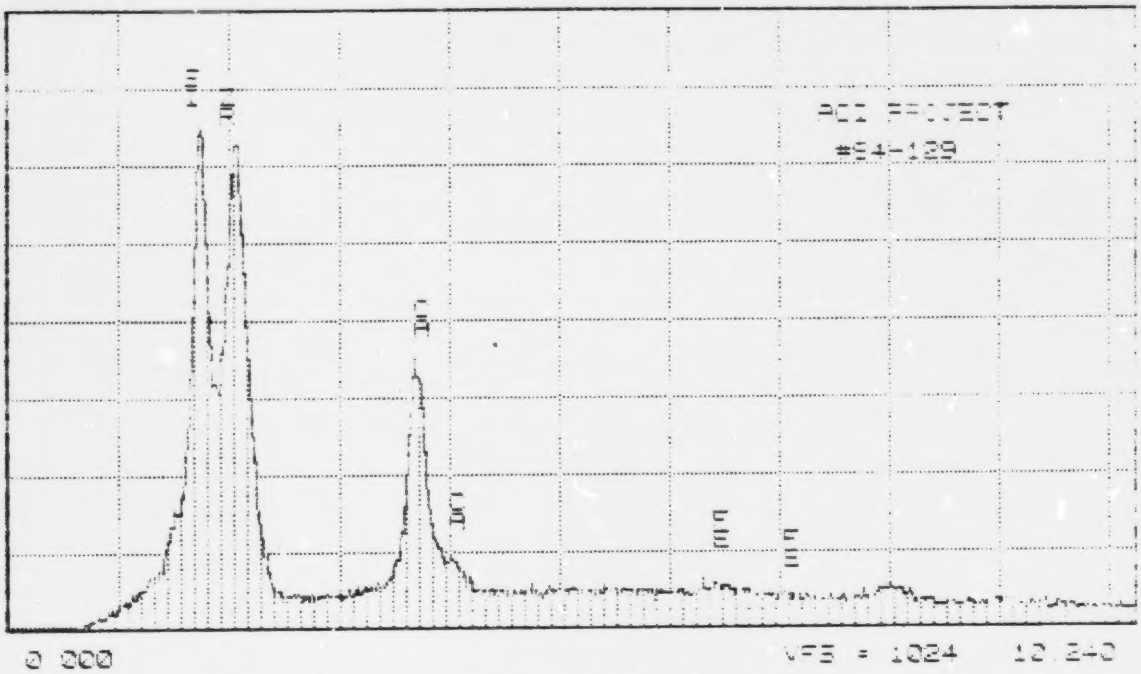


10 DUST STONE DUST SAMPLE FROM CBCRL

11

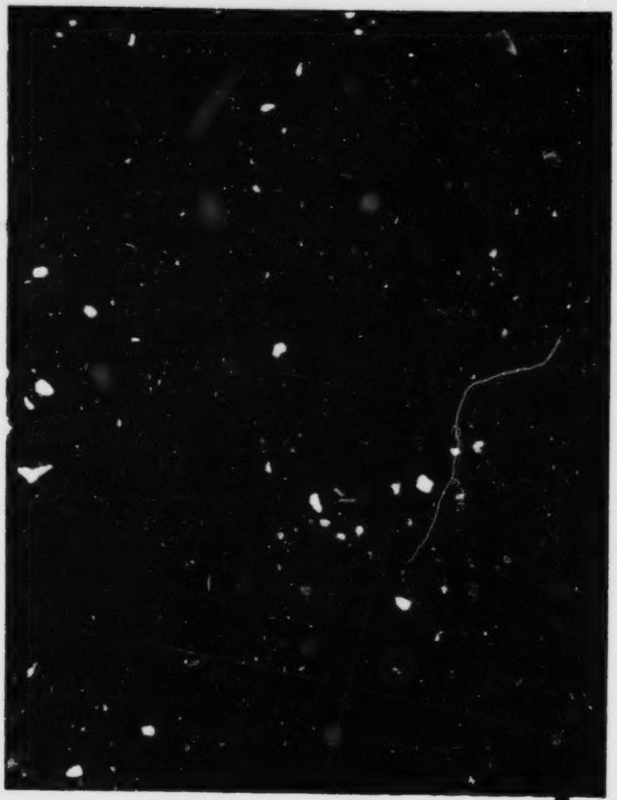
2a. PHOTOMICROGRAPHS: N.B.S. STANDARDS

1. NBS SRM 1878 respirable alpha quartz quantitative Xray Powder Diffraction Standard: 95.5 ± 1.1 wt.% crystalline quartz. Equivalent spherical diameters: 0.33-5 um. Mass mean equivalent spherical diameter: 1.62um. This sample was dry dispersed on Nuclepore (0.1 um pores). We carbon coated for imaging at 20kv, (BEI x1000). Note the agglomeration and flocculated appearance.

2. NBS SRM 2679a (BI) quartz on filter media: 30.8 ∓ 3.4 ug of SRM1878 quartz mixed with 370 ug flint clay (SRM 97a). The mixture was prepared in 0.5% aqueous gum tragacanth solution and dried onto a cellulose acetate membrane filter (0.45 um pores). This photomicrograph shows the contact area between loaded sample (dark) and filter medium (light). Note the large particulate size range due to agglomeration and the masking effect of the gum tragacanth solution. SEI x 750 carbon coated, 20kv.

3. NBS SRM 2679a (BI) filter background. SEI x10,000, 20kv. carbon coated.

4. NBS SRM 2679a (BI) particulates. Note the masking effect of the gum solution. SEI x2000, carbon coated.



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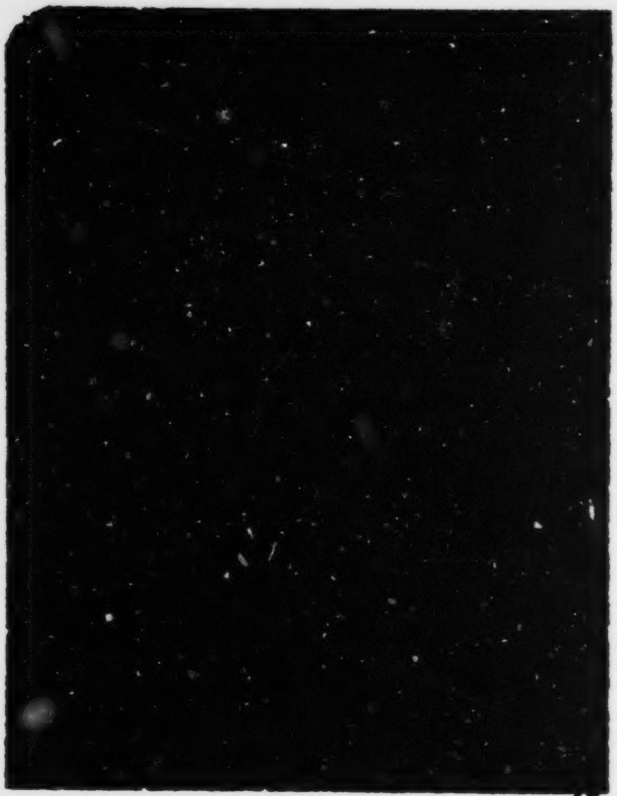
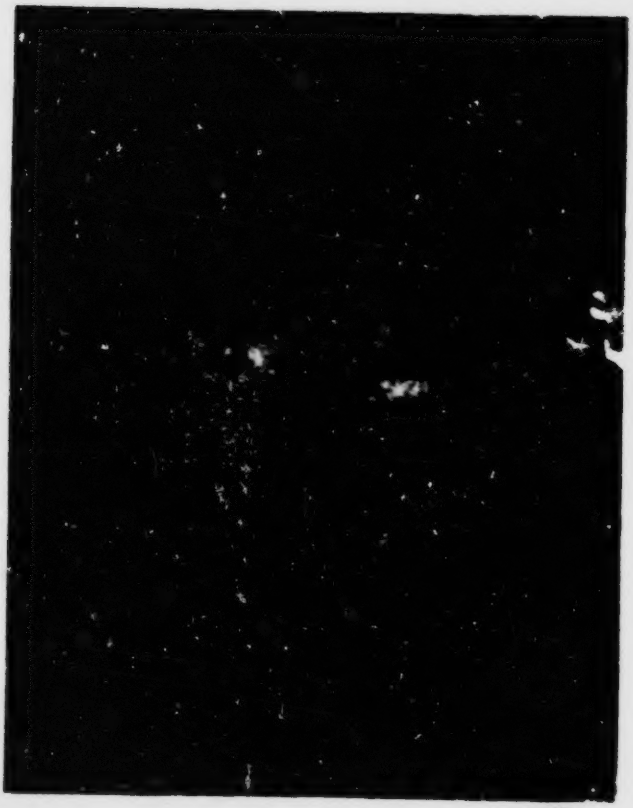


2



3

4

2b. EDX SPECTRA: NBS STANDARDS

1. NBS SRM 1978 alpha quartz powder dispersed on Nuclepore. Particle spot analysis shows only silica present, ie. SiO2. 10 second count.

2. NBS SRM 2679a (B1): quartz on cellulose acetate membrane filter (0.45 um pore size). Filter BI contains a 30.8 ± 3.4ug quartz to 370 ug clay mixture in a 0.5% aqueous gum tragacanth solution. The area raster (EDX #2) shows a complex spectrum dominantly clay: Si, Al, Ca, Ti, Cl, K. The percentage of quartz cannot be assertained since the clay contains some 20% silicon. 100 seconds count.

3. NBS SRM2679a (B1): Spot analysis on particles. The spectrum suggests kaolinite clay, but the presence of quartz in an agglomerate cannot be ruled out.

4. NBS SRM 2679a (B1): Accessory particle spot analysis: a contaminant? 10 second count.

5. NBS SRM 2679a (B1): Background spectrum (30 second count). The large chlorine and calcium peaks account for the complexity of the area raster (EDX #2).

ATLANTIC COAL INSTITUTE MON 08-APR-85 [illegible]

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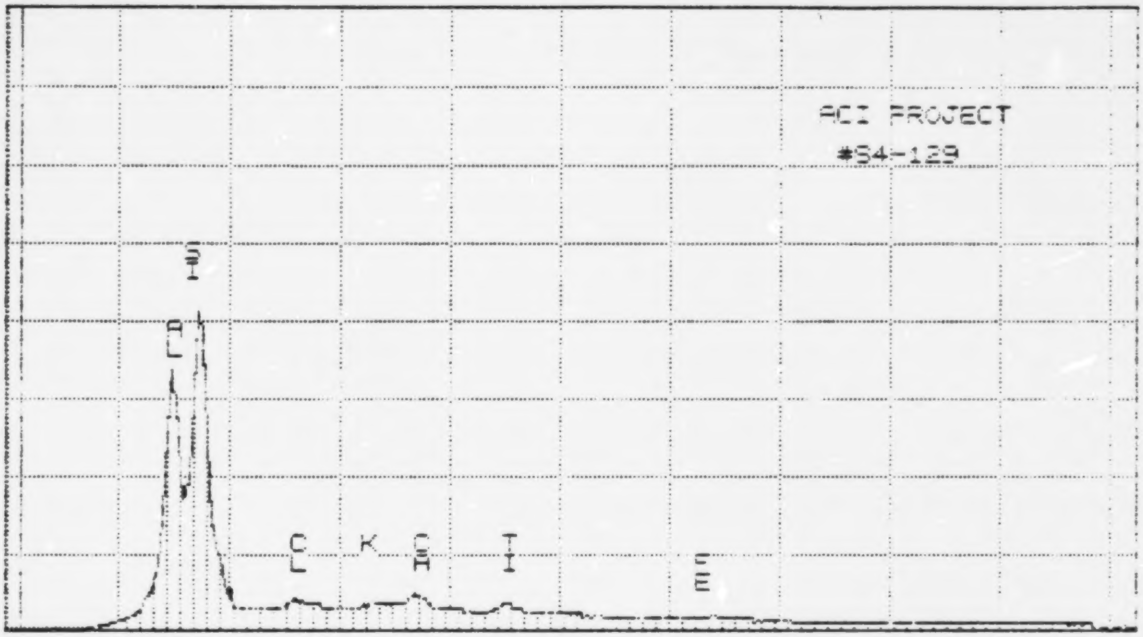

0.000 VFS = 16384 10.240

100 DUST NBS RESP QUARTZ ON FILTER AREA SCAN

2

ATLANTIC COAL INSTITUTE SAT 06-APR-85 15:07

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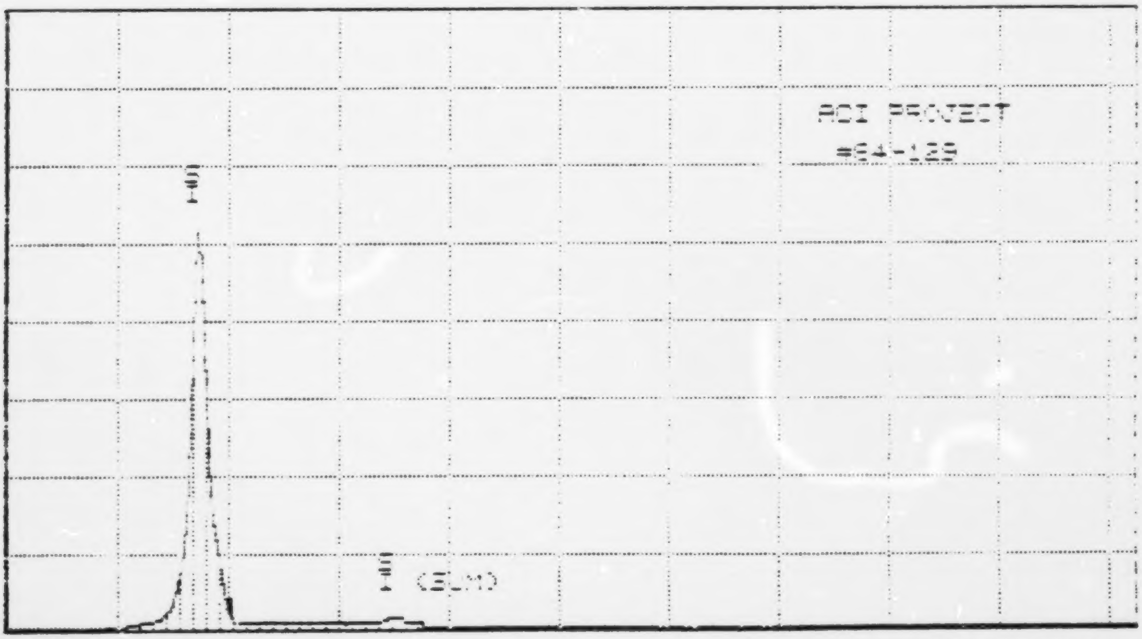

1

0.000 VFS = 16384 10.240

DUST NBS RESPIRABLE ALPHA QUARTZ STANDARD

ATLANTIC COAL INSTITUTE MON 15-APR-85 11:20
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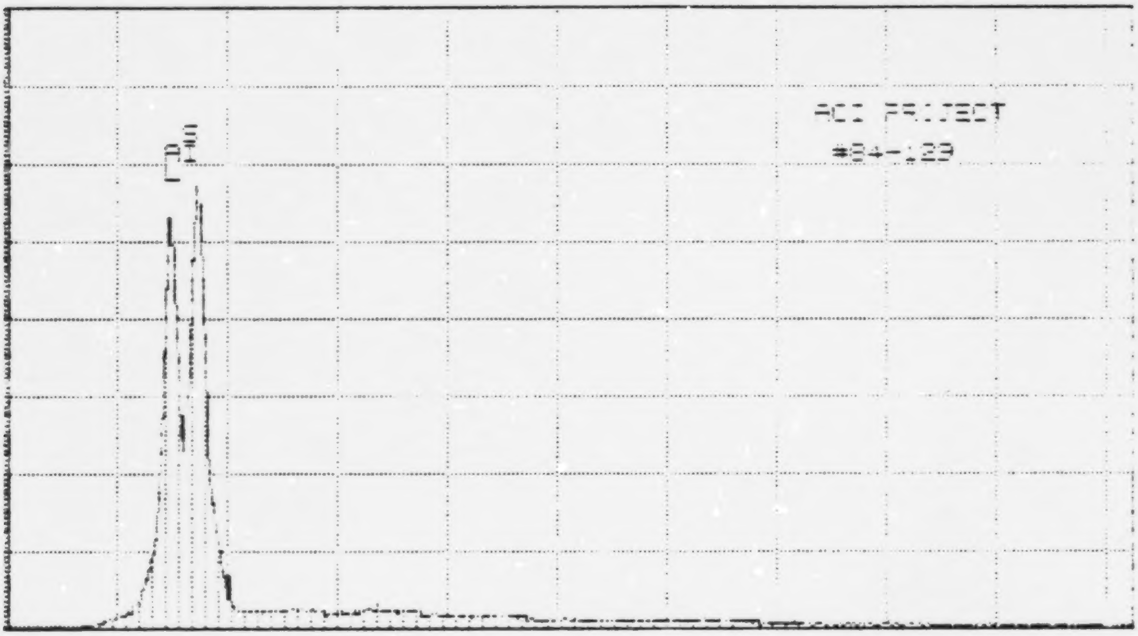

0.000 VFS = 2048 10.240
10 DUSTS NBS QUARTZ ON FILTER

3

ATLANTIC COAL INSTITUTE MON 15-APR-85 11:13
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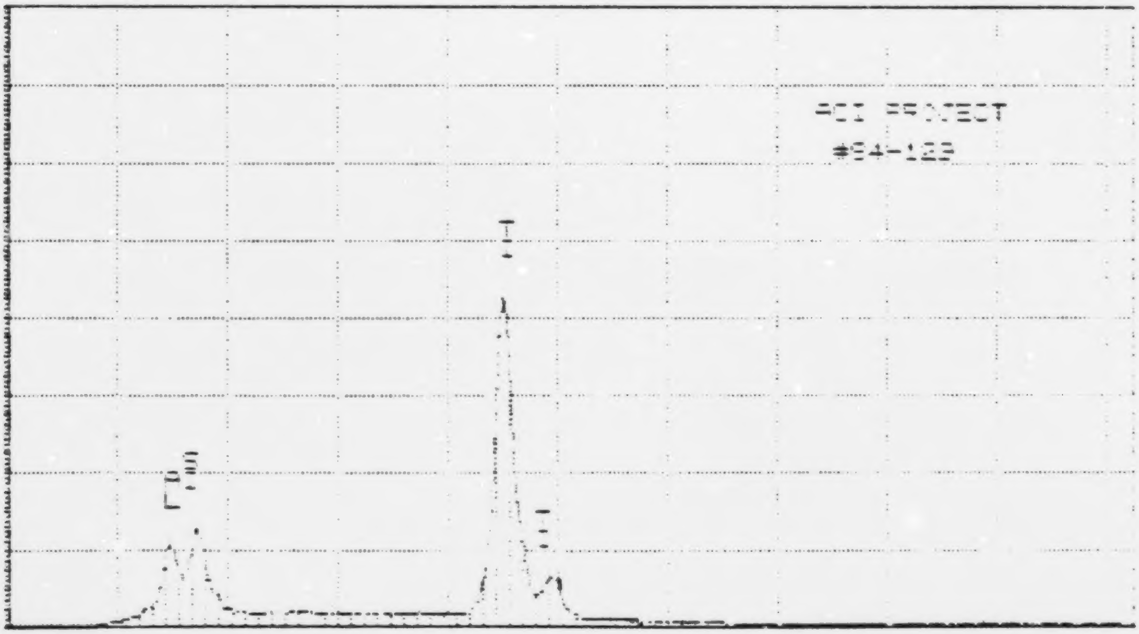

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10 DUSTS NBS QUARTZ ON FILTER

4

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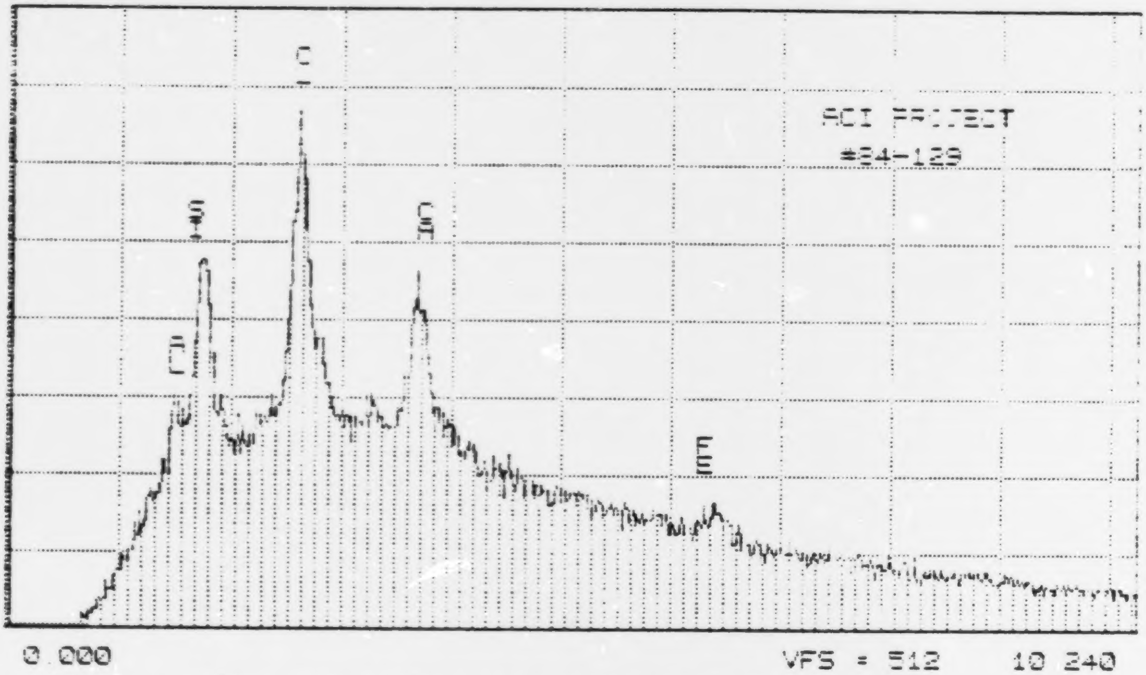


5

30 DUSTS NBS QUARTZ ON FILTER BACKGROUND

2c. PHOTOMICROGRAPHS
CHEMPLEX MINERALOGICAL POWDERS*

1. Quartz Standard, 20kv, BEI, x500. Backscattered image shows quartz as medium grey and contaminant particles (of greater average atomic number) as brighter (EDX 8-10)

2. Biotite Standard. 20kv, BEI, x500. Note various shades of grey; the largest particles are all biotite but vary in average atomic number. (EDX #1-3).

3. Siderite Standard. gold coated, 20kv, SEI, x5000 (EDX 4-7).

4. ibid BEI. Siderite particles ($FeCO3$) appear bright in relation to the mounting substrate and most contaminant particles are darker.

5. ibid. SEI, x2000 quartz particle EDX #7.

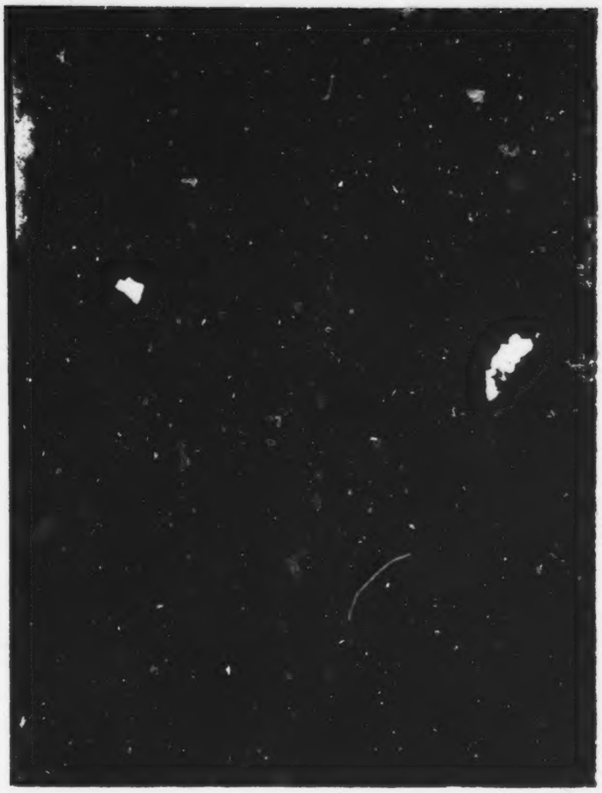
6. Microcline Standard, 20kv, BEI, x1000 (EDX 12-15).

7. ibid. composite particle barite + microcline.

8. Calcite Standard, 20kv, BEI, x750 (EDX 16-21).

*Powders dispersed on Nuclepore filters and carbon coated, except where noted.

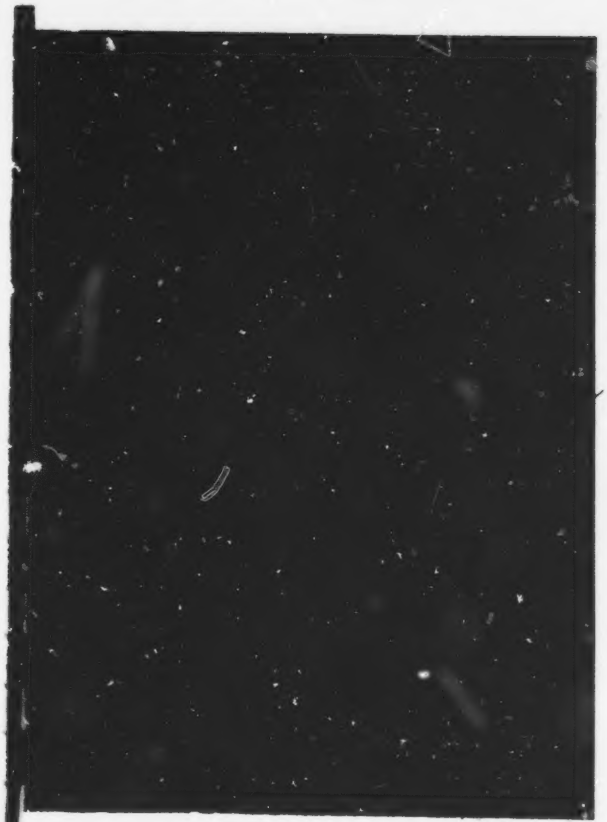
1



2

3

4



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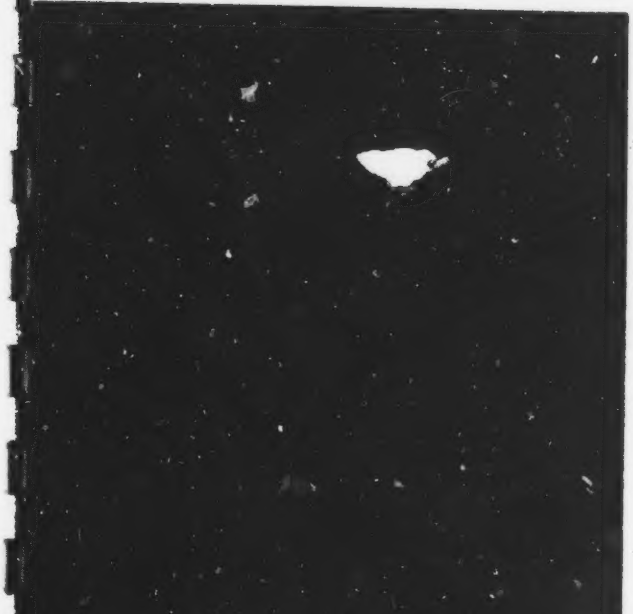
6



7

8

2d. EDX SPECTRA
CHEMPLEX MINERALOGICAL POWDERS

1. Biotite Standard. [K(Mg, Fe)3 ($AlSi_3O_{10}$) (OH)2]. Typical spectrum: Different particles will show spectral differences due either to geometry effects or elemental substitution. This and subsequent spectra are based on 10 second counts at 20kv.

2. Contaminant particle in biotite standard. iron silicate mineral (?).

3. ibid (photo #2) Barite

4. Siderite Standard ($FeCO_3$) typical spectrum, gold coated showing traces of Mg, Mn, and Si.

5. Contaminant particle in Siderite Standard, Galena (?).

6. ibid. clay mineral (kaolinite?)

7. ibid photo #5. quartz grain.

8. Quartz Standard (SiO_2) typical spectrum.

9. Contaminant particle in quartz standard, possibly zincite (ZnO) with manganese substitution.

10. Contaminant in Quartz Standard, possibly chromite ($FeCr_2O_4$).

11. Microcline Standard [$k(AlSi_3O_8)$] typical spectrum.

12. Contaminant particle in Microcline Standard photo #7 (bright spot). Barite.

13. ibid. siderite (?).

14. ibid. rare earth mineral (?), possibly monazite.

15. ibid. plagioclase (?)

16. Calcite Standard (CaCO3) typical spectrum.

17. Contaminant particle in Calcite Standard. Likely Scheelite ($CaWO_4$).

18. ibid. Rare earth mineral, possibly monazite.

19. ibid. siderite (?)

20. ibid. Fe silicate.

21. ibid. unknown contaminant

Note: All samples are powders dispersed on Nuclepore filters and carbon coated except where noted.

ATLANTIC COAL INSTITUTE WED 03-APR-85 19:07

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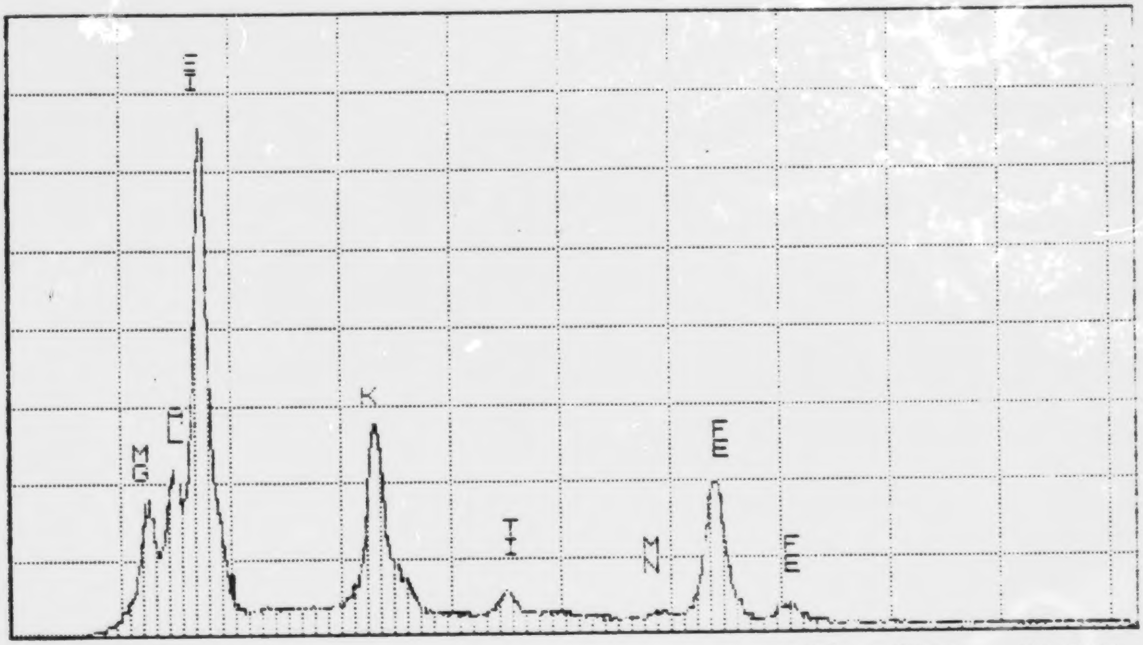

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10 DUSTS BIOTITE STANDARD PARTICLE 1

1

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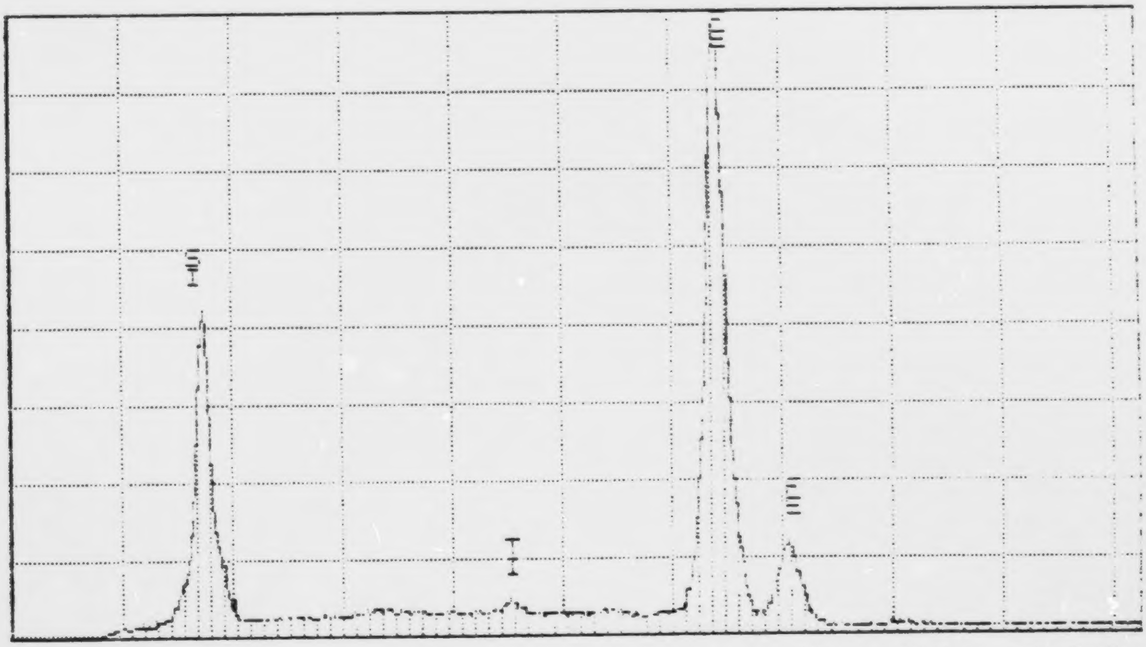

0.000 VFS = 2048 10.240

10 DUSTS BIOTITE STANDARD PARTICLE 2

2

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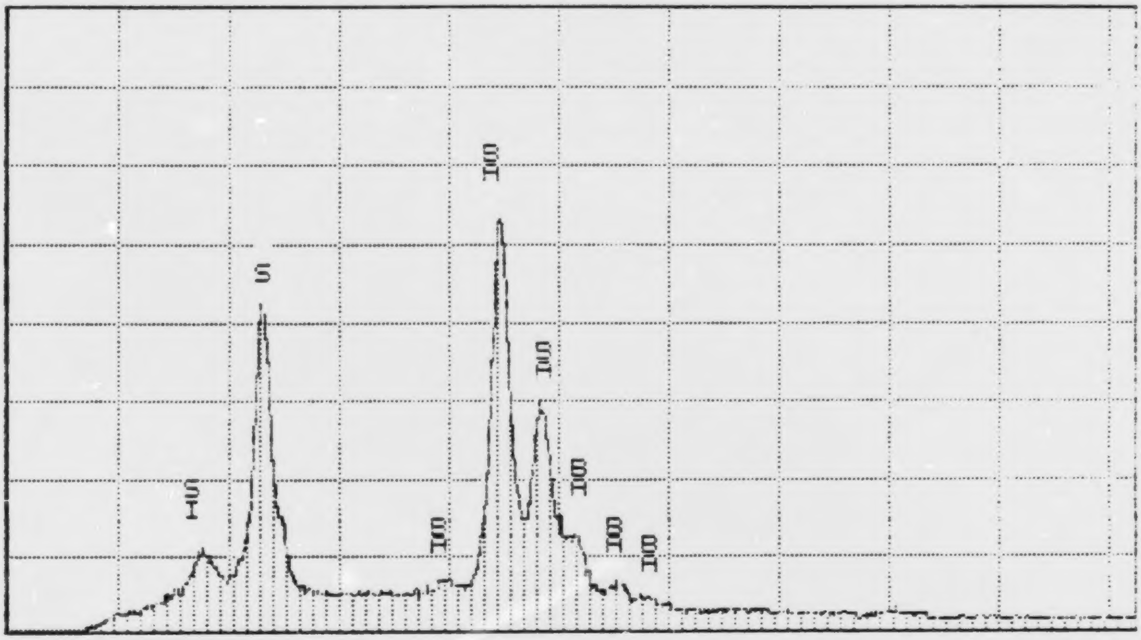

3

10 [illegible] BIOTITE STANDARD PARTICLE 2

ATLANTIC COAL INSTITUTE   WED 10-APR-85 14:07

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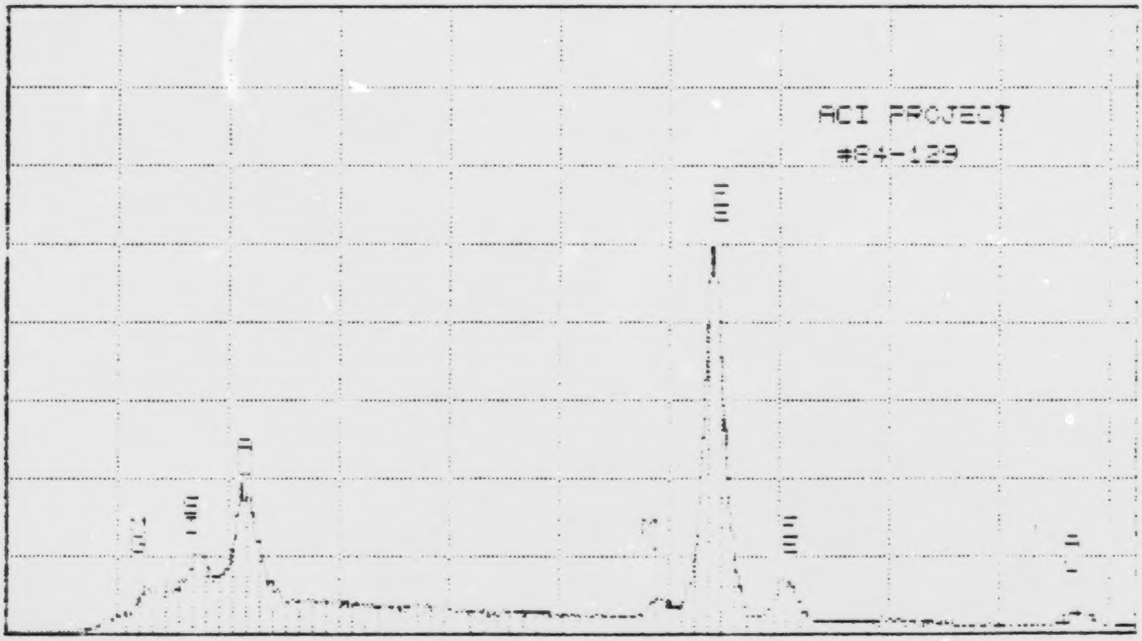

4

10   DUST SIDREITE STANDARD

+2409

ATLANTIC COAL INSTITUTE MON 08-APR-85 14:45

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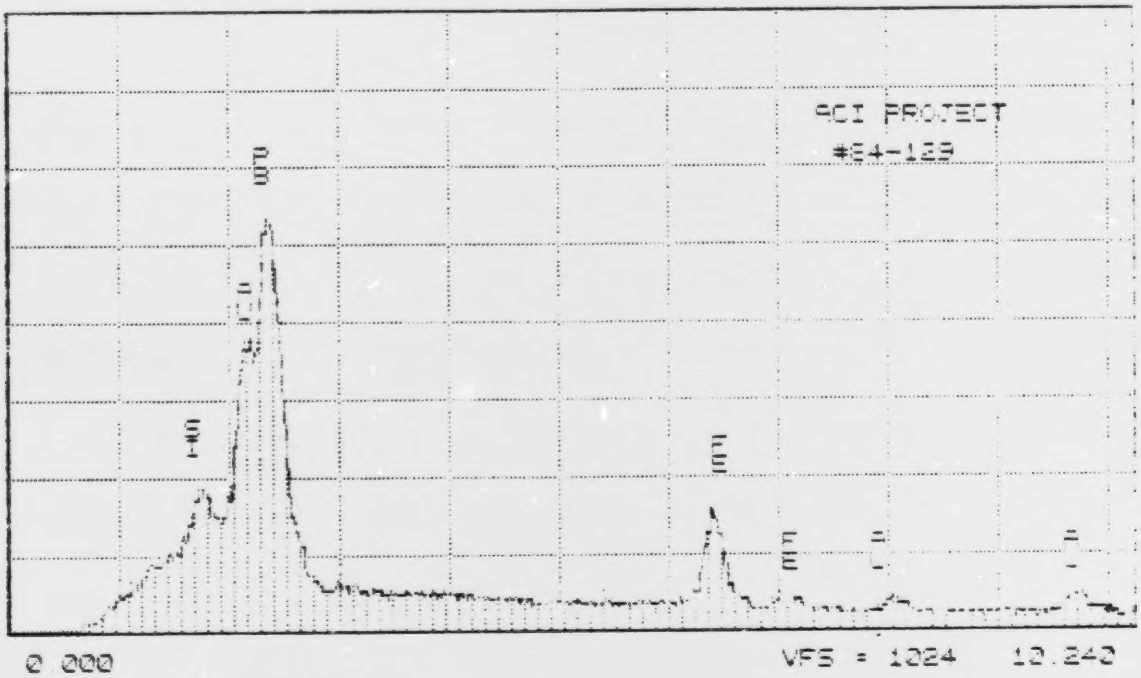

10 DUST SIDERITE STANDARD

5

N= +2410

ATLANTIC COAL INSTITUTE MON 08-APR-85 14:47

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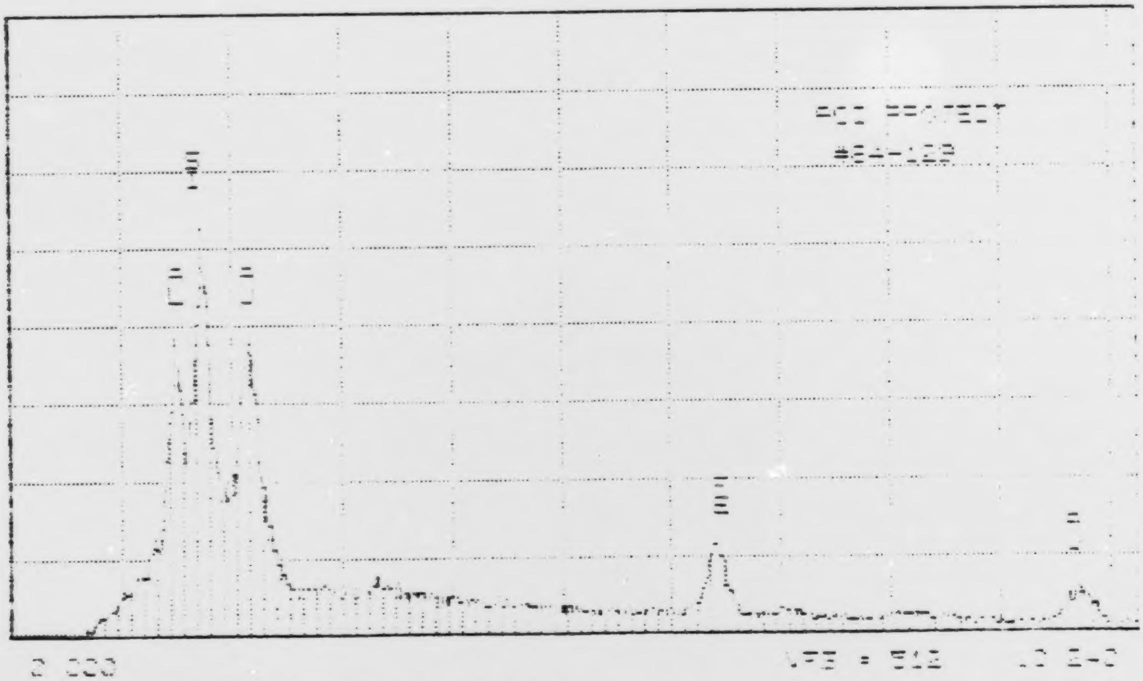

10 DUST SIDERITE STANDARD

6

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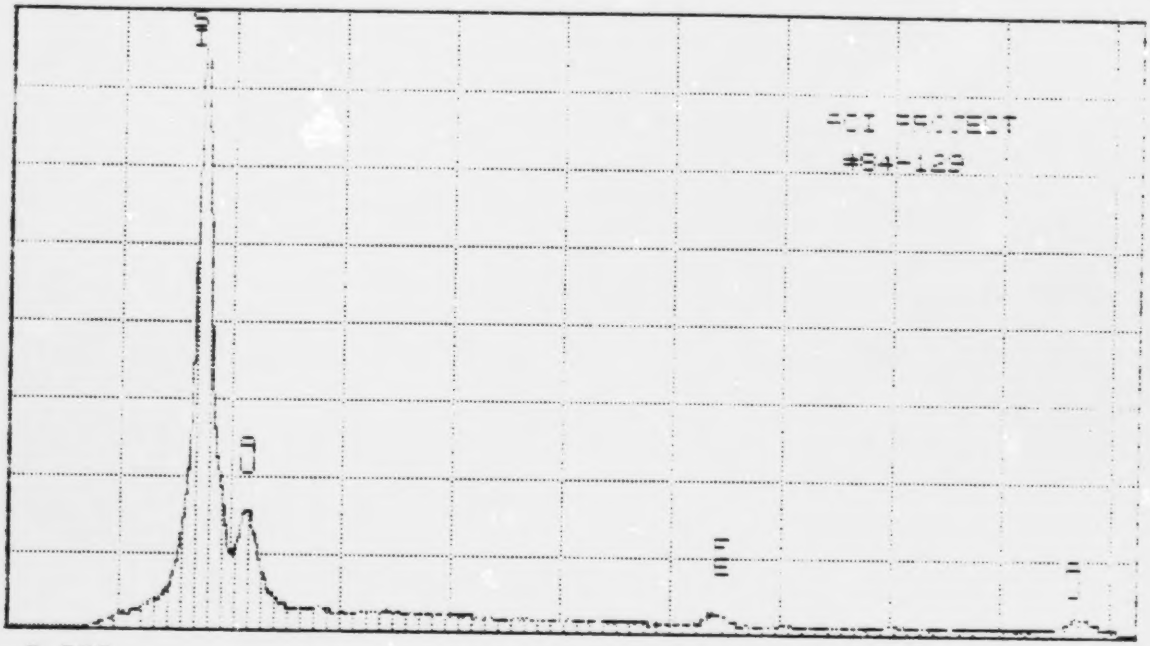

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10 DUST SIDERITE STANDARD

7

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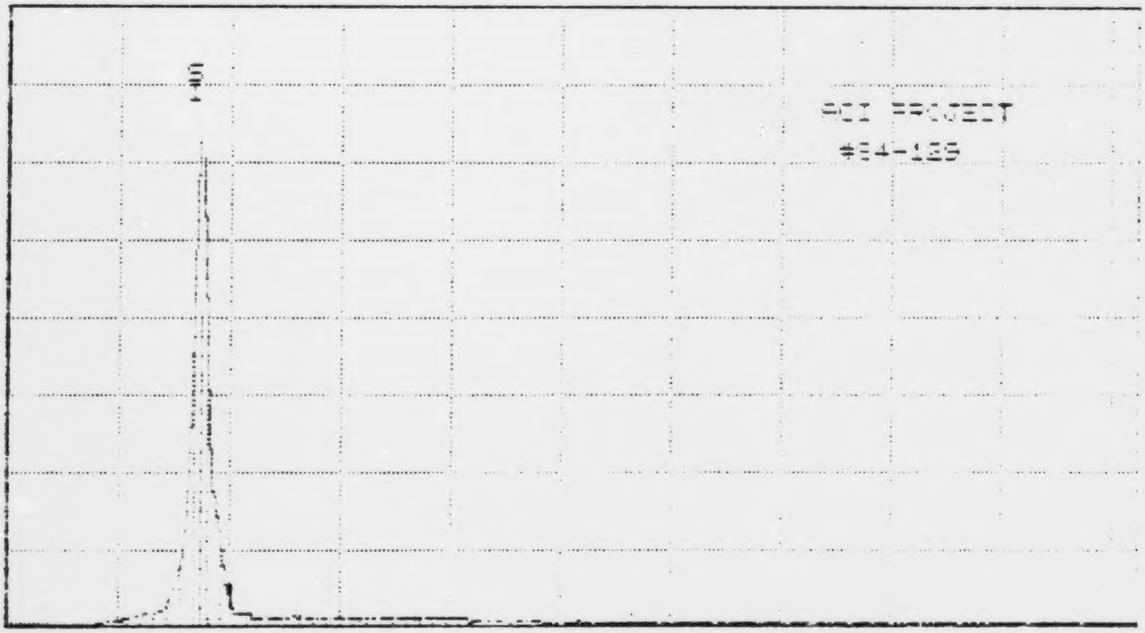
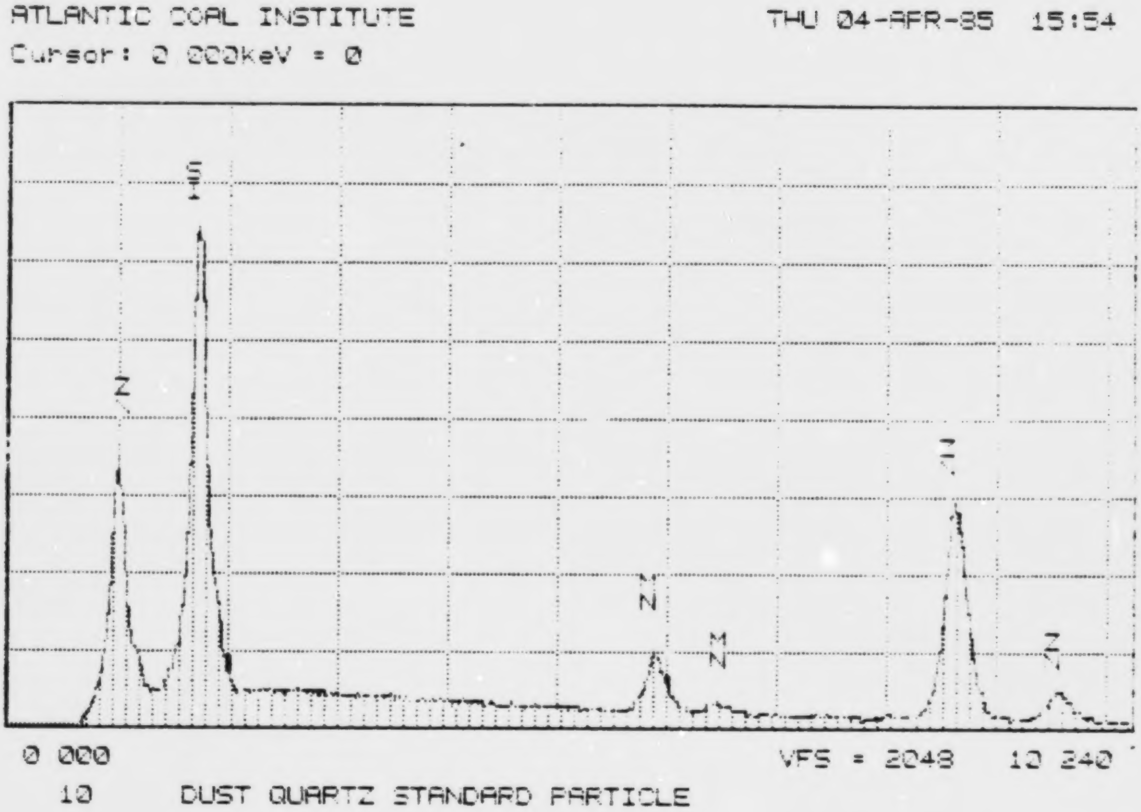


0.000 SI-14 VFS = 2048 10.240

10 DUST QUARTZ STANDARD

8



ATLANTIC COAL INSTITUTE THU 04-APR-85 15:54
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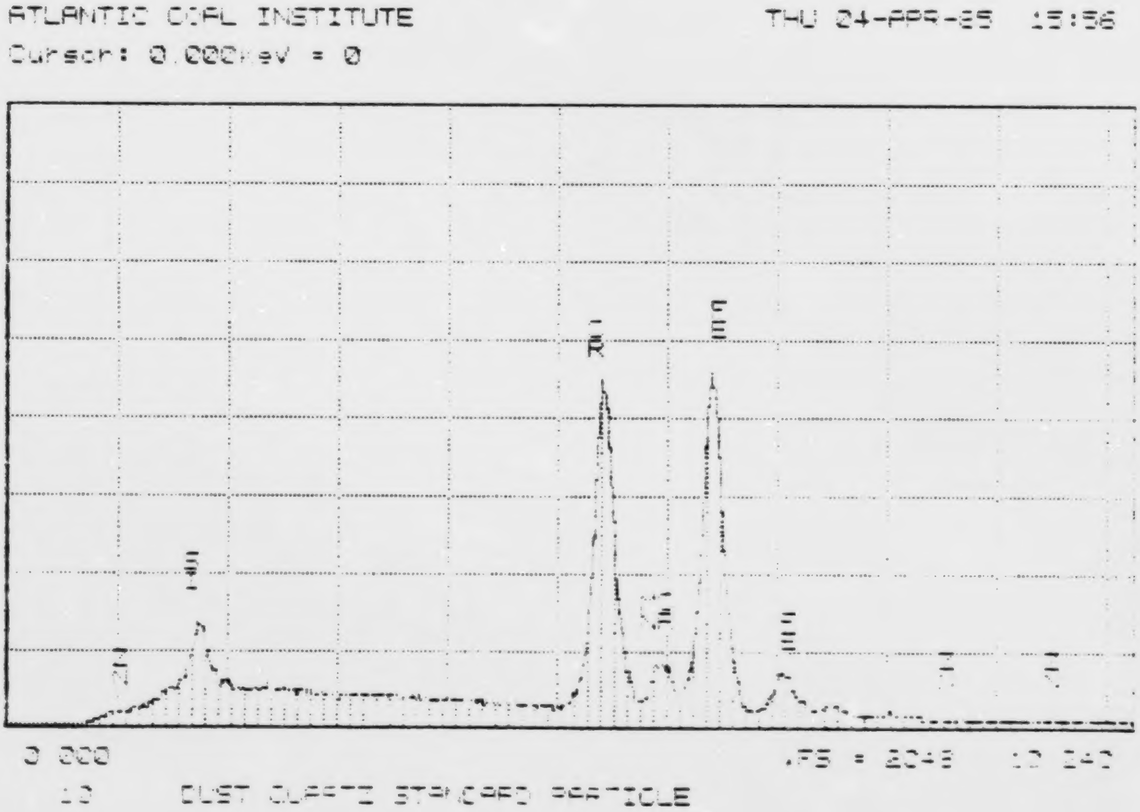
0.000 VFS = 2048 10.240
10 DUST QUARTZ STANDARD PARTICLE

9



ATLANTIC COAL INSTITUTE THU 04-APR-85 15:56
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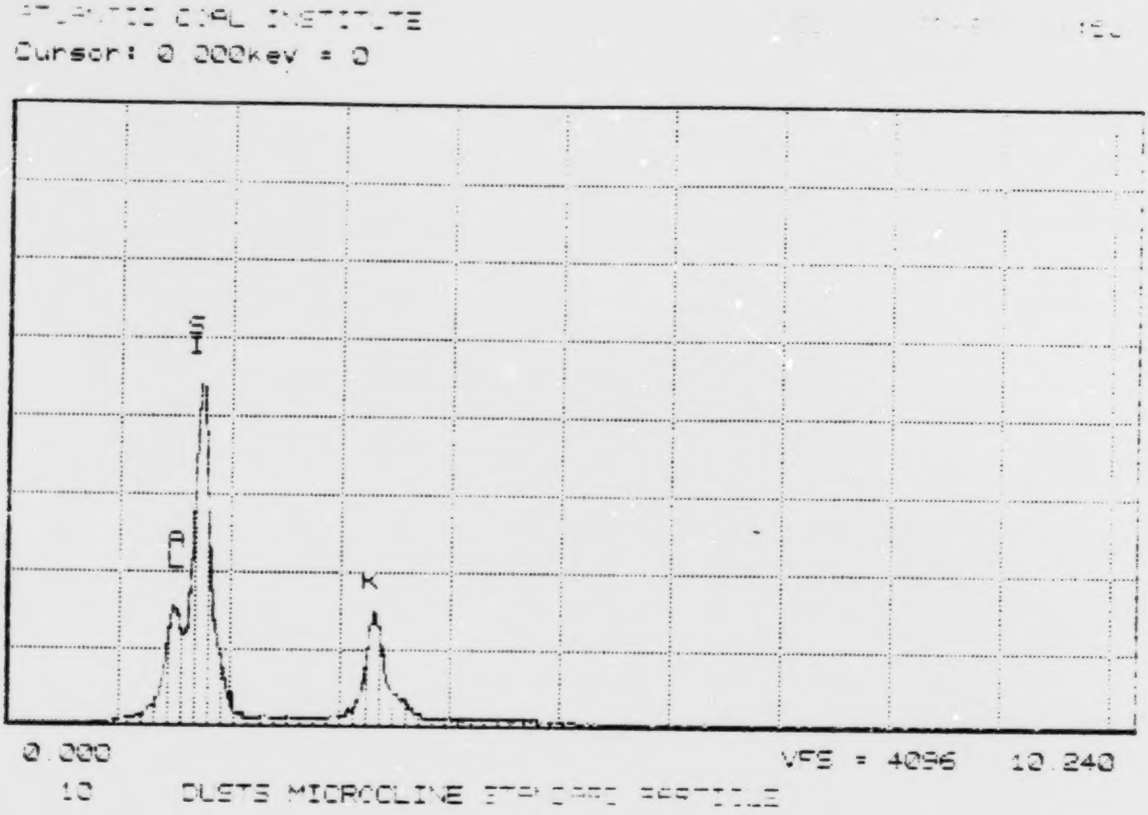
0.000 VFS = 2048 10.240
10 DUST QUARTZ STANDARD PARTICLE

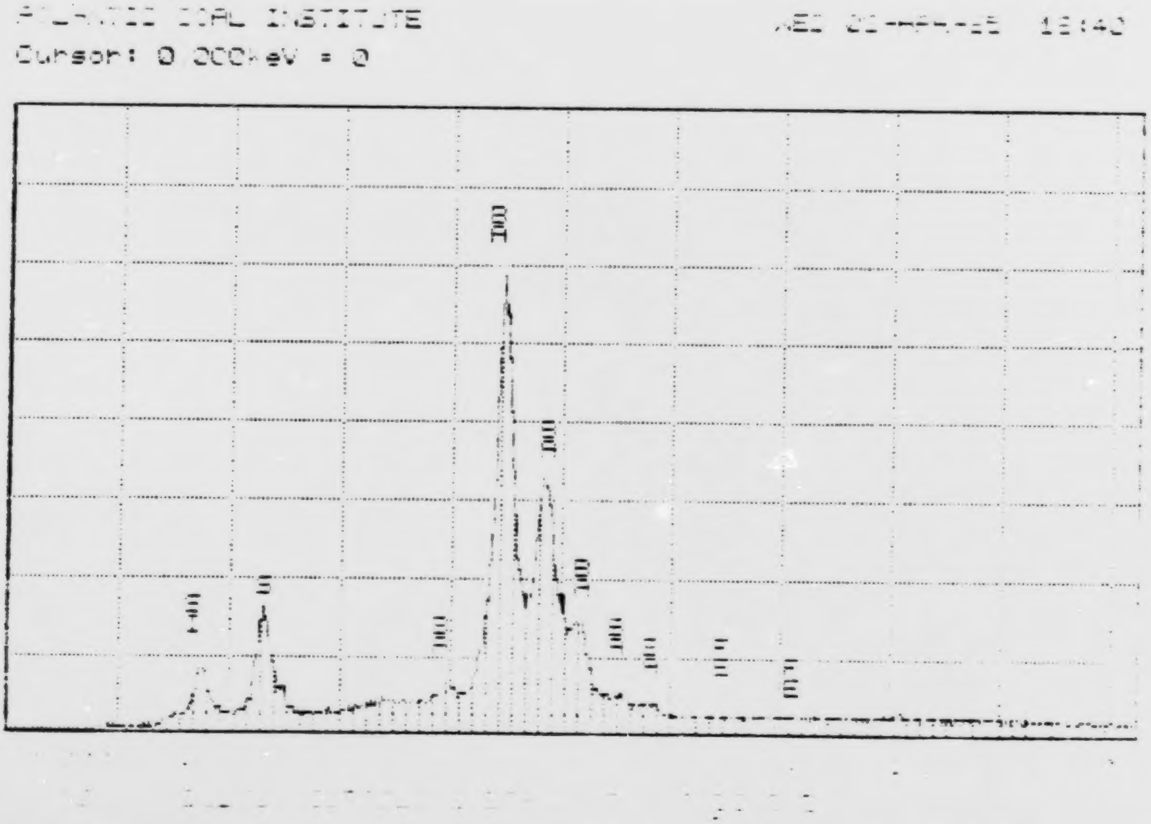
10


ATLANTIC COAL INSTITUTE

Cursor: 0.000keV = 0

10 DUSTS MICROCLINE STANDARD PARTICLE

11


ATLANTIC COAL INSTITUTE

WED [illegible] 13:40

Cursor: 0.000keV = 0

12

ATLANTIC COAL INSTITUTE WED 03-APR-85 18:42
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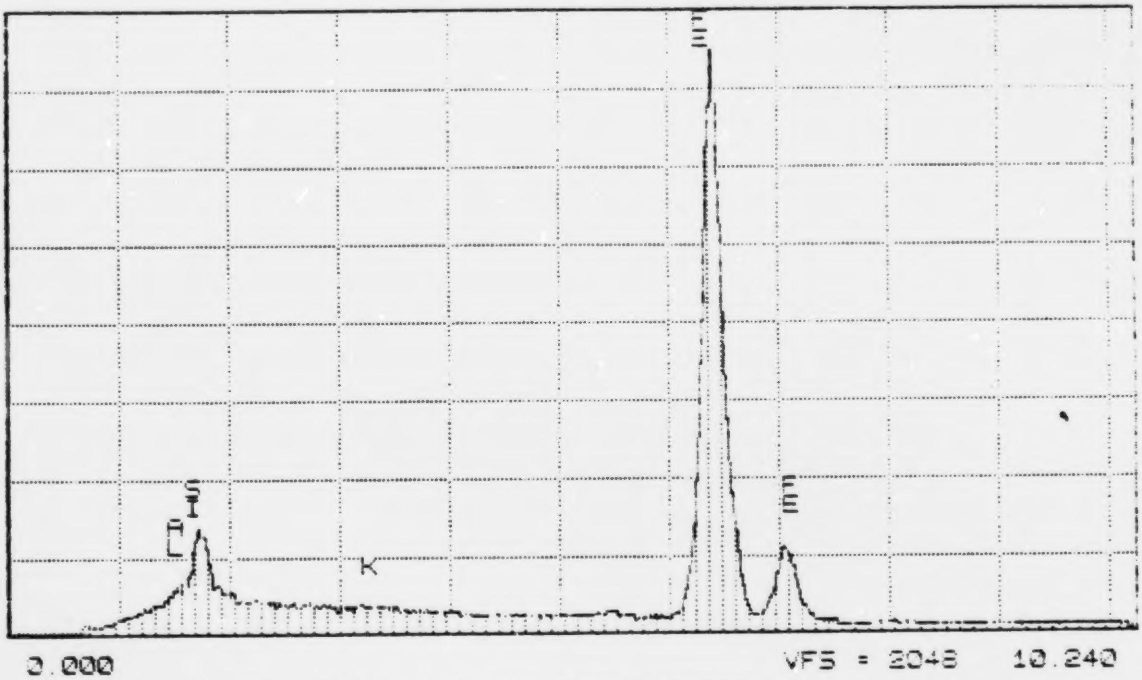

10 DUSTS MICROCLINE STANDARD PARTICLE 3

13

ATLANTIC COAL INSTITUTE WED 03-APR-85 18:44
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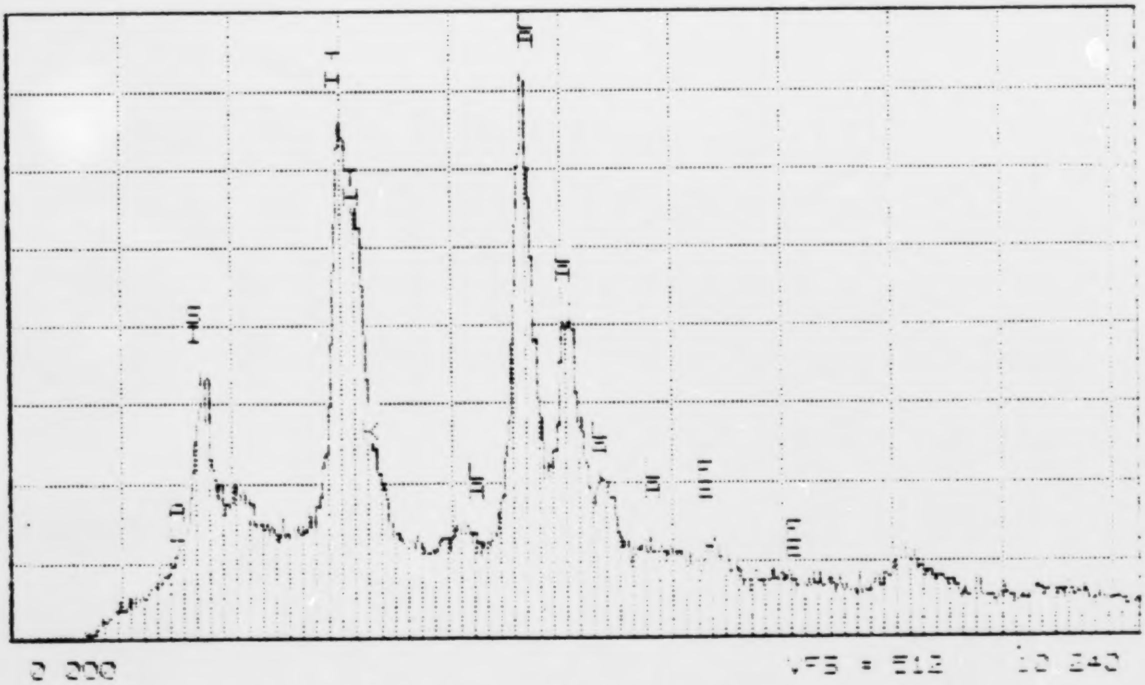
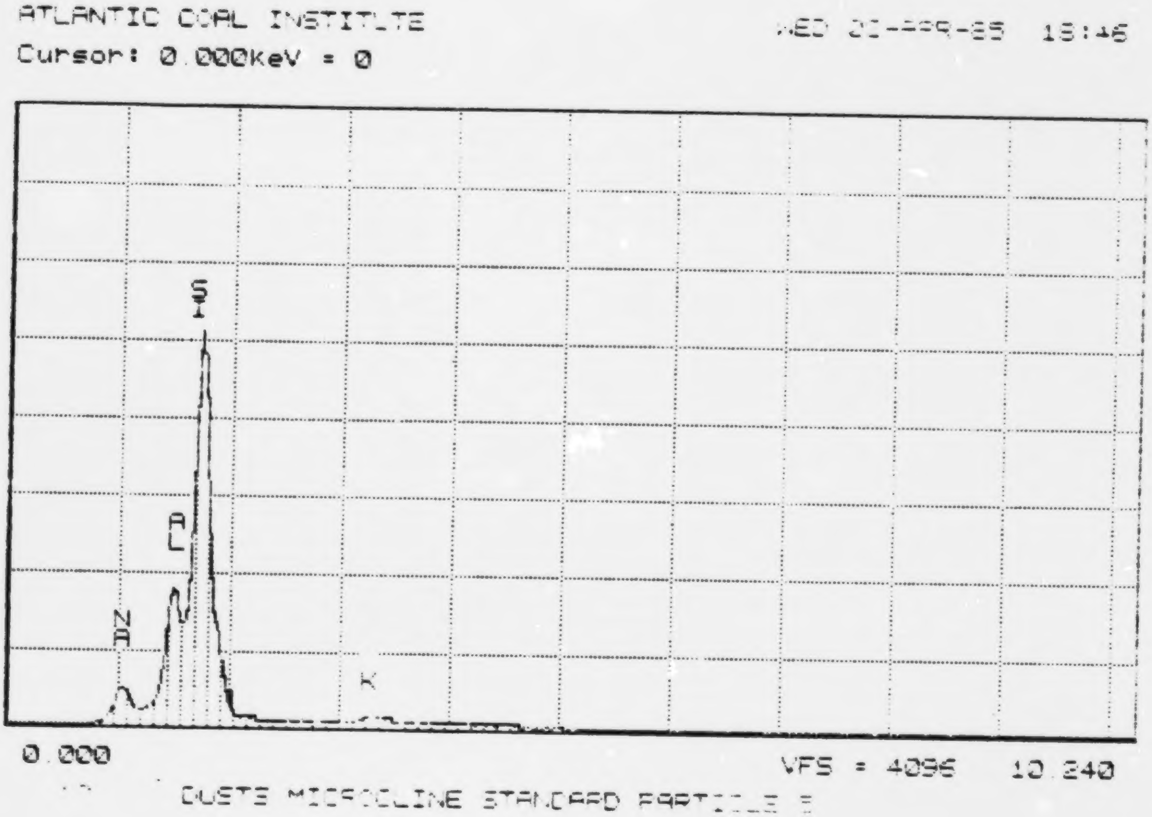


10 DUSTS MICROCLINE STANDARD PARTICLE 4

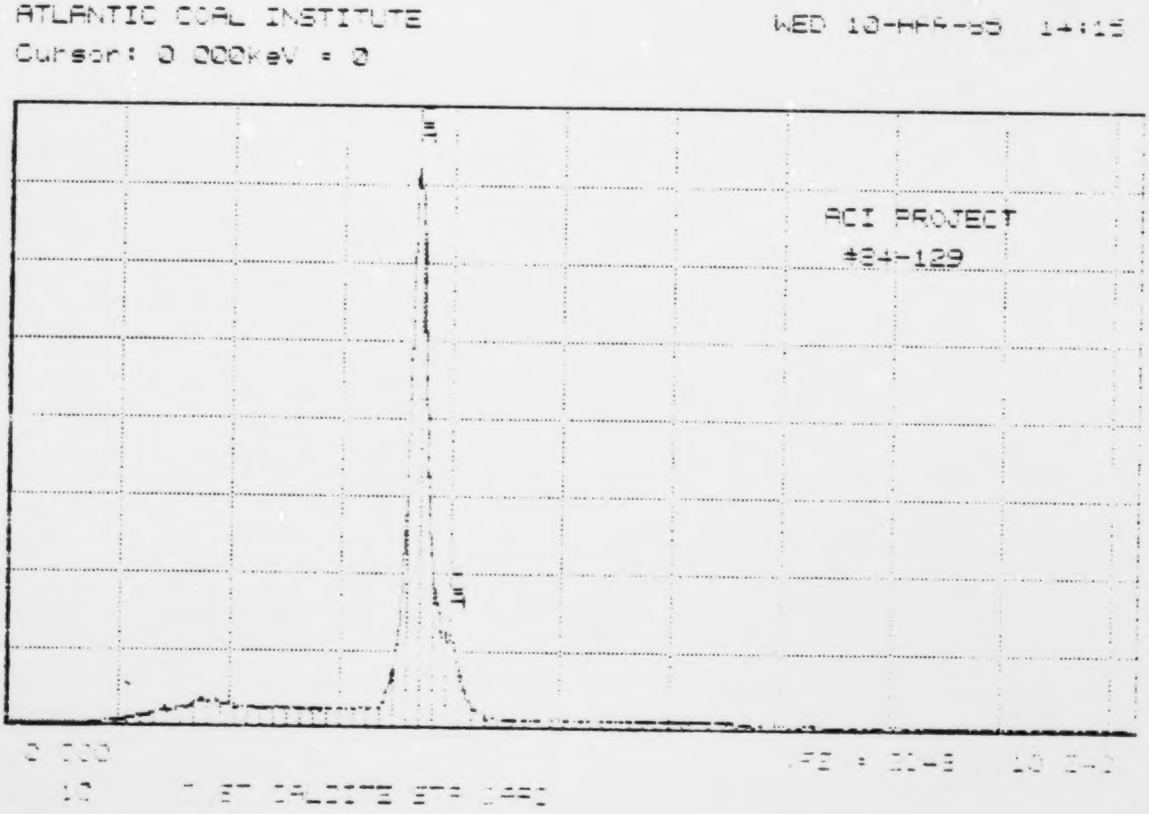
14



ATLANTIC COAL INSTITUTE
WED 24-APR-85 15:46
Cursor: 0.000keV = 0

0.000
VFS = 4096 10.240
DUSTE MICROCLINE STANDARD PARTICLE 3

15



ATLANTIC COAL INSTITUTE
WED 10-APR-85 14:15
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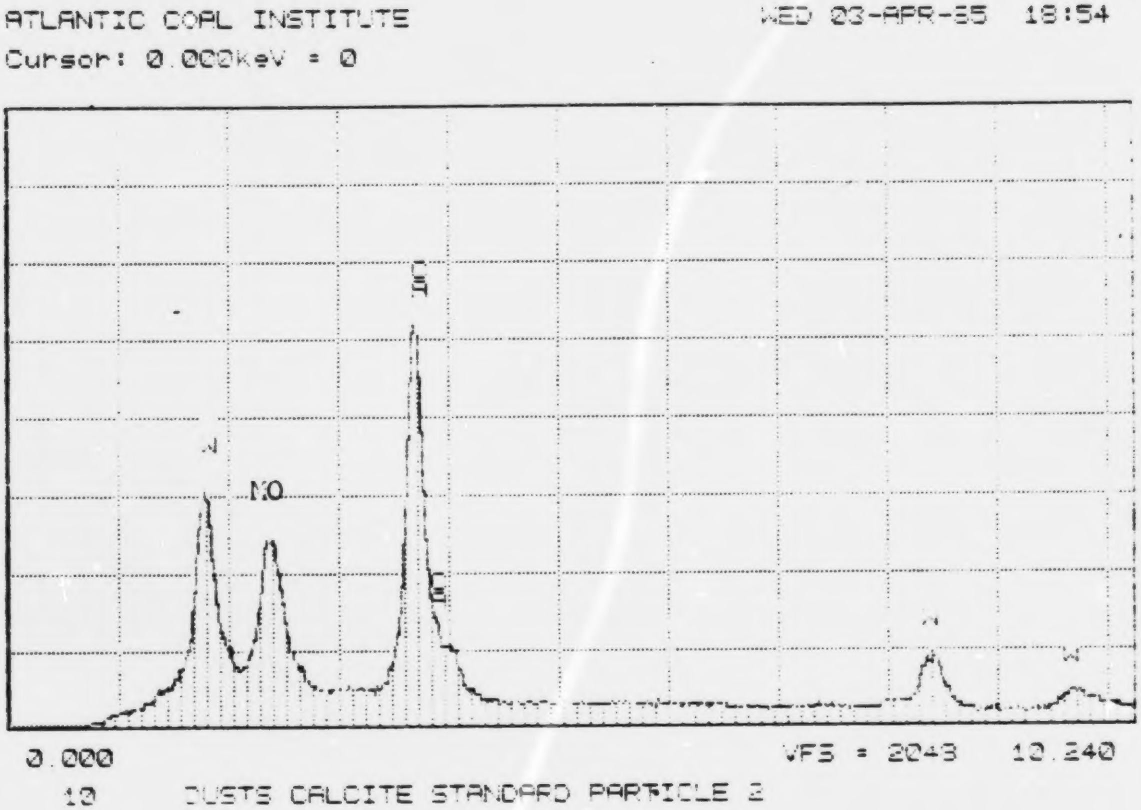
ACI PROJECT
#84-129

0.000
VFS = 2048 10.240
10 TEST CALCITE STANDARD

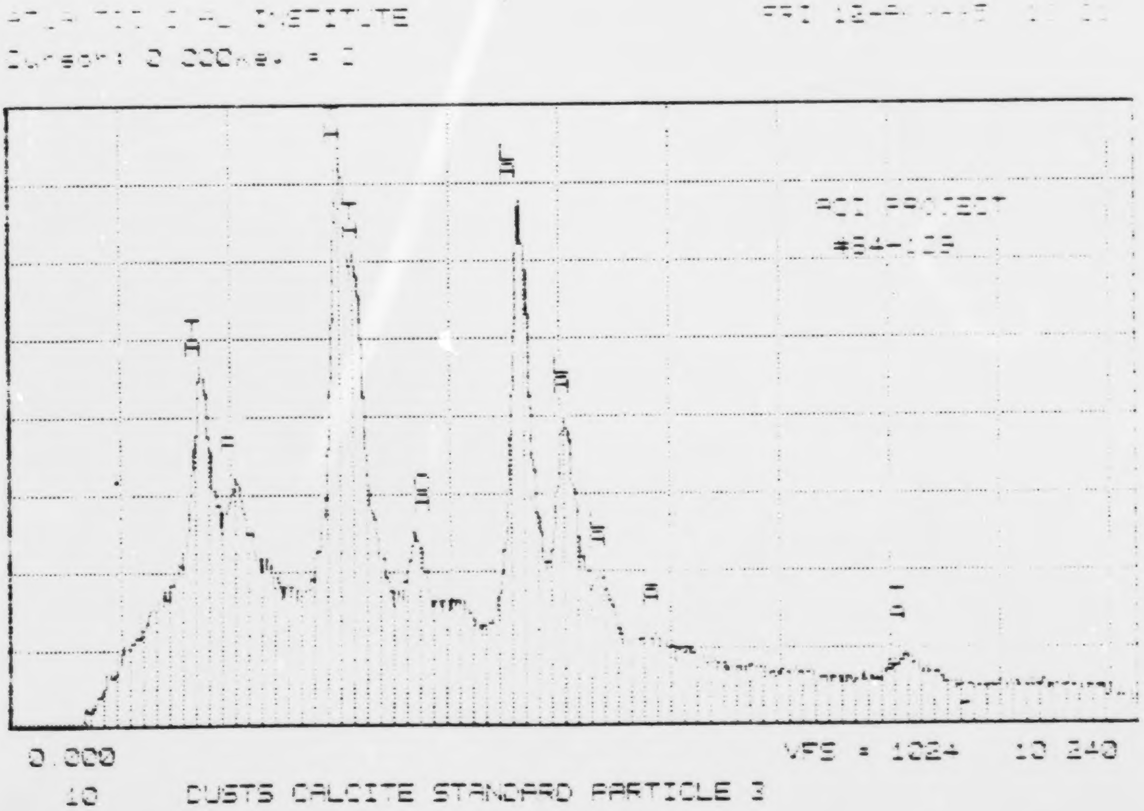
16


ATLANTIC COAL INSTITUTE WED 03-APR-85 18:54

Cursor: 0.000keV = 0

10 DUSTS CALCITE STANDARD PARTICLE 2

17


ATLANTIC COAL INSTITUTE FRI 12-APR-85 [illegible]

Cursor: 0.000keV = 0

ACI PROJECT
#84-103

10 DUSTS CALCITE STANDARD PARTICLE 3

18

ATLANTIC COAL INSTITUTE WED 03-APR-85 19:03

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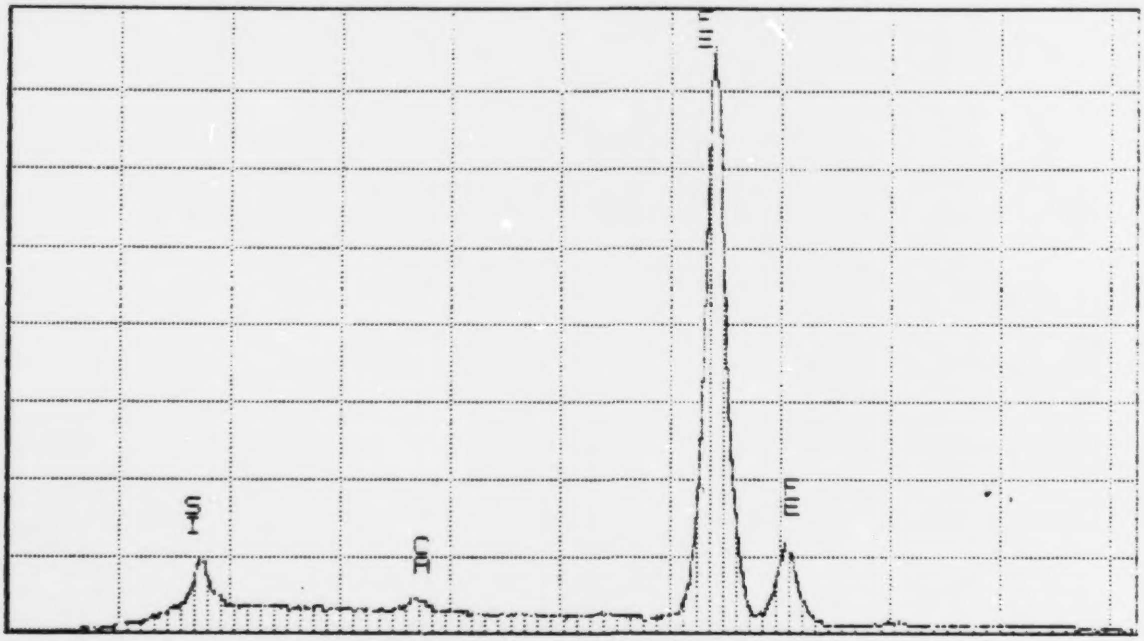

0.000 VFS = 4096 10.240

10 DUSTS CALCITE STANDARD PARTICLE 5

19

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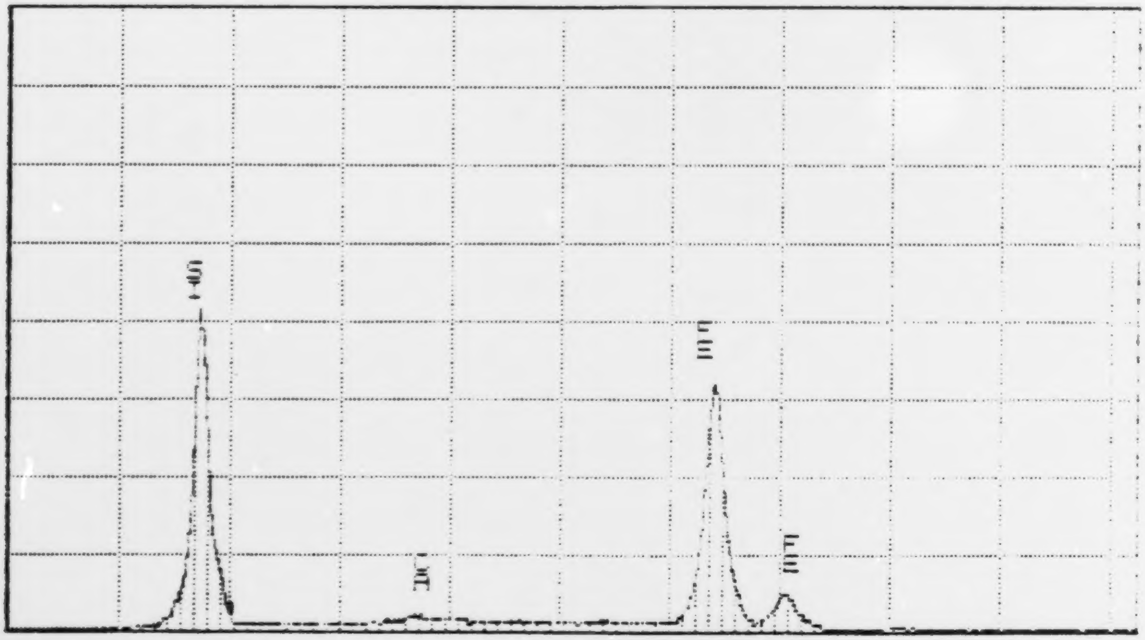
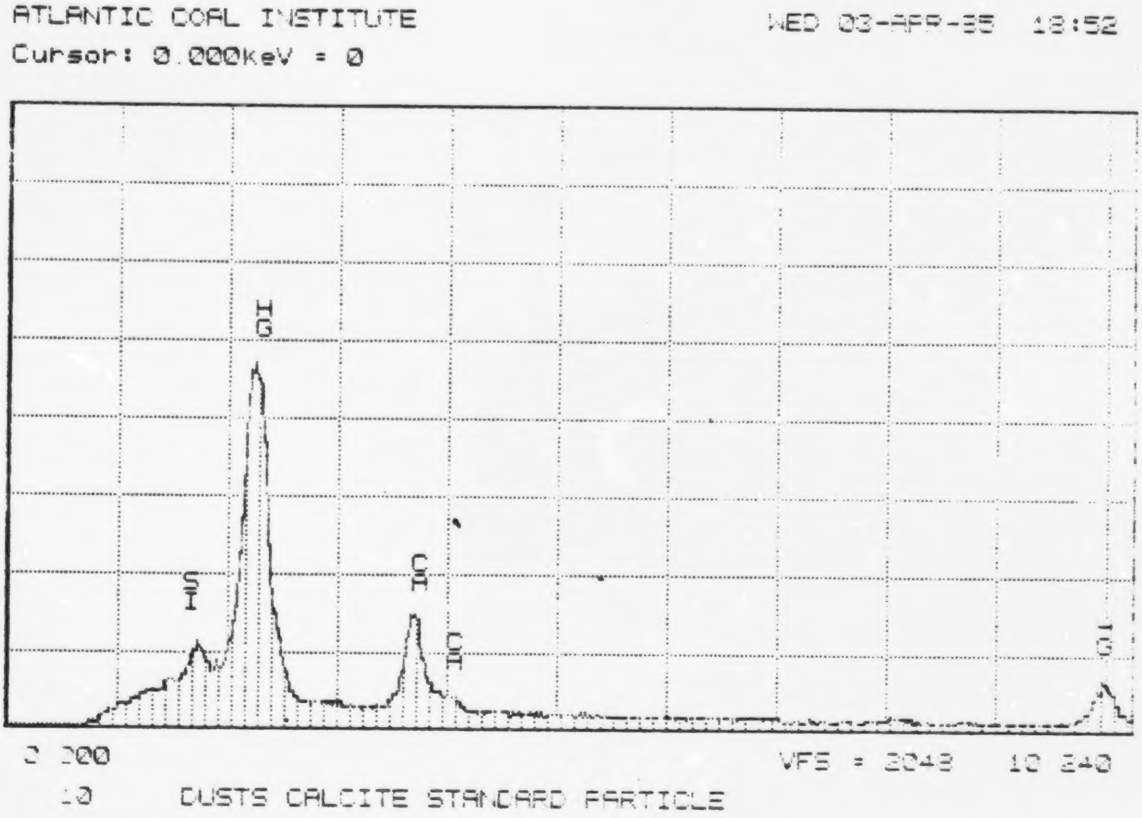


0.000 VFS = 4096 10.240

10 DUSTS CALCITE STANDARD PARTICLE 6

20



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0.200 VFS = 2048 10.240

10 DUSTS CALCITE STANDARD PARTICLE

2e. EDX SPECTRA: ASTIMEX MICROPROBE MINERAL STANDARDS

1. EDX spectrum for calcite, $CaCO_3$, showing a single peak of Ca. Elements C and O are not reported by the Edx system because of a beryllium window shielding the Si(Li) detector. Analysis is based on a spot mode of 50 second count at 20kv. The following chemistry is reported by Astimex in weight %:

C	O	Mg	Ca	Mn
12.02%	47.98	0.01	39.98	0.01

Note that we did not detect Mg or Mn with our EDX system indicative of concentrations below our analytical threshold.

2. EDX spectrum, as above, for Fluorite: CaF_2

F	Ca
48.67%	51.33

3. Magnetite: Fe_3O_4

O	Cr	Fe
26.67%	0.14	72.19

Note: No Cr detected.

4. Haematite: Fe_2O_3

O	Mg	Al	V	Fe
30.06%	0.03	0.10	0.04	69.86

Note: Al, Mg, V not detected

5. Galena: PbS

S	Pb
13.40%	86.60%

6. Rutile: TiO_2

O	V	Ti	Fe
40.35%	0.38	58.59	0.66

Note: Fe, V not detected

7. Quartz: SiO_2

O	Si
53.26%	46.74

8. Albite: NaAlSi3O6

O	Na	Al	Si	K	Ca
48.76%	8.60	10.34	32.03	0.18	0.09

Note the attenuated Na peak because of our beryllium window. Also, we do not detect K or Ca.

9. Plagioclase: (Ca,Na) Al(Al, Si) Si2O6

O	Na	Mg	Al	Si	K	Ca
47.08%	3.23	0.08	15.10	25.34	0.34	8.43

Ti	Fe	Sr	Ba
0.04%	0.29	0.07	0.01

The following elements are not detected: mg, K, Ti, Sr, Ba.

10. Dolomite: MgCa(CO3)2

C	O	Mg	Ca	Mn	Fe
13.03%	52.06	13.18	21.73	0.02	0.02

Elements Mn and Fe are not detected.

11. Apatite: Ca5(PO4)3(OH)

H	O	P	Ca
0.20%	41.41	18.50	39.89

Neither O nor H can be detected by the EDX system because of the beryllium window.

12. Anhydrite: CaSO4

O	S	Ca
47.07%	23.55	29.38

13. Barite: BaSO4

O	S	Ba
27.42%	13.74	58.84

14. Monazite: (Ca, La, Y, Th) PO4

O	P	Ca	La	Ce	Pr	Nd	Th
25.9%	12.1	0.4	12.9	30.8	4.2	8.7	3.8

Note that Ca, Pr, and Nd are not recognized in the EDX spectrum due to the extensive peak overlaps.

15. Chlorite: $Mg6\ AlSi_3O_{10}(OH)_8$

H	C	O	F	Mg	Al	Si	Ca
1.54%	0.02	51.14	0.02	20.21	9.57	14.04	0.02

Cr	Fe	Ni
0.67	2.57	0.19

Note that we detected Cr but missed the low Z elements (H,C,O) and F, Ca, Ni.

16. Biotite: $K(Mg, Fe)3\ AlSi_3O_{10}(OH)_2$

H	O	Mg	Al	Si	K	Ca
0.46%	43.95	11.77	8.01	18.10	8.23	0.07

Ti	Mn	Fe
1.06	0.03	8.33

Note that Mn, Ca and light elements are omitted.

17. Sphalerite:

S	Fe	Zn	Sn
33.38%	0.10	65.68	0.45

Elements Fe and Sn are not labeled but longer counting statistics would likely have detected them.

18. Marcasite: FeS2

Fe	S
46.54%	53.46

ATLANTIC COAL INSTITUTE WED 06-FEB-85 15:42

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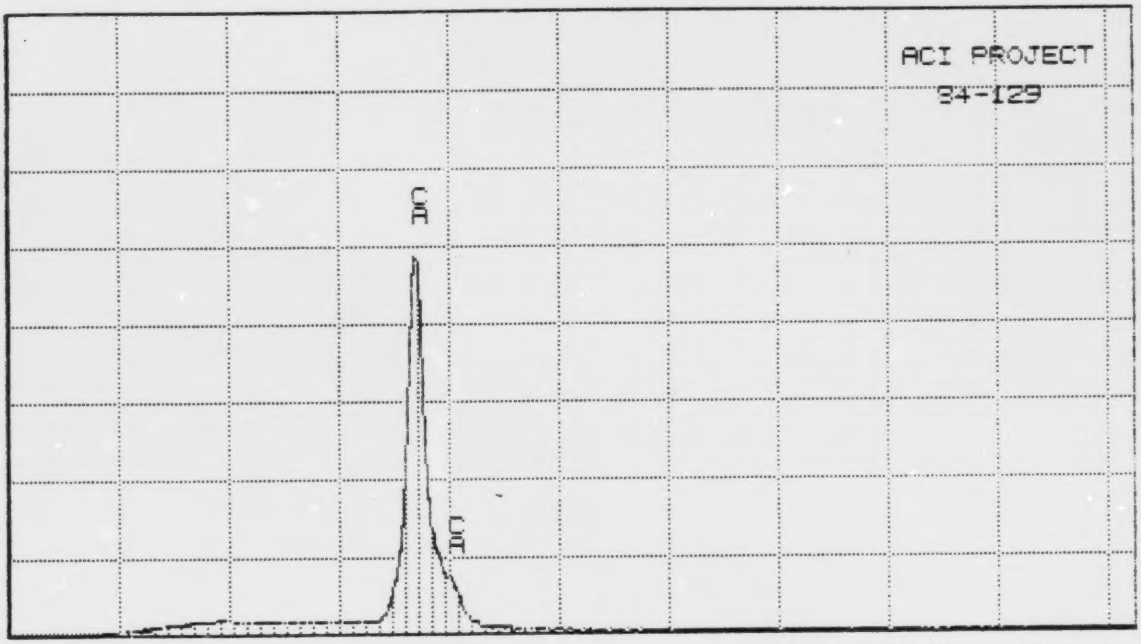

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50 MIN STD. CALCITE 20KV SPOT

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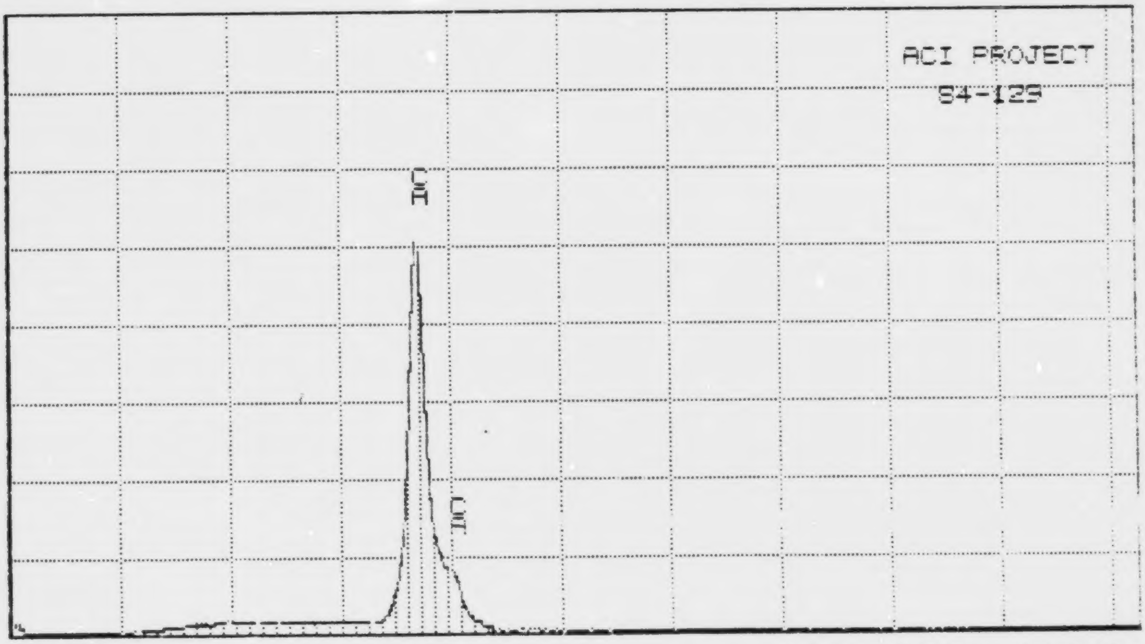

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50 MIN STD. FLUORITE 20KV SPOT

2

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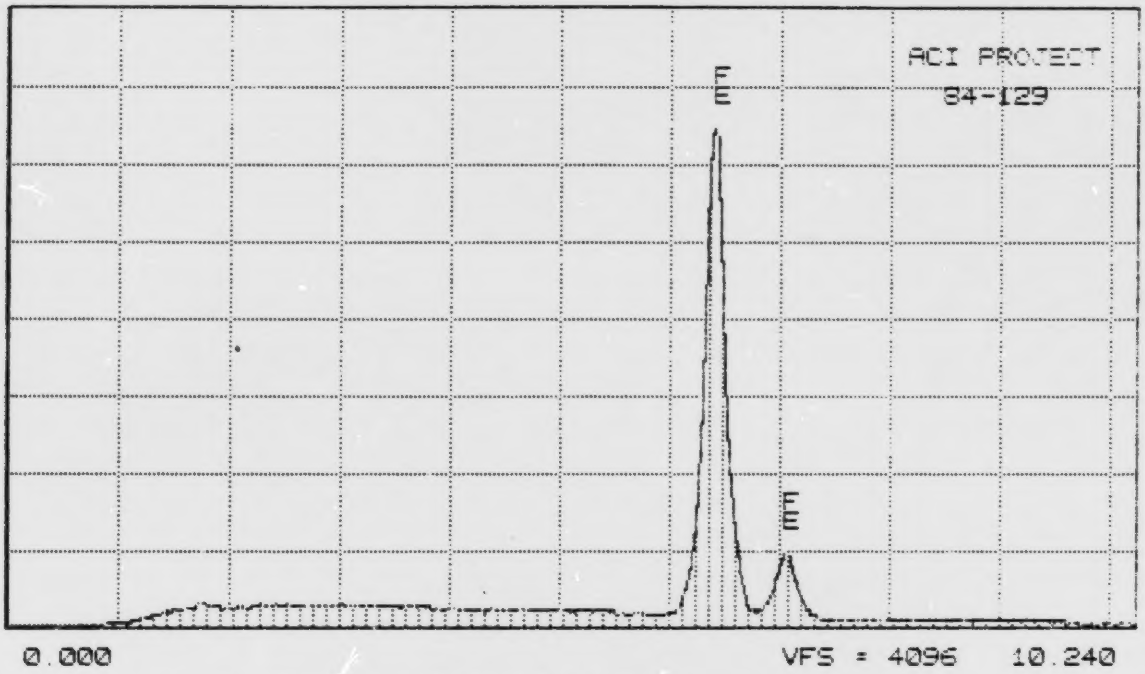

50   MIN STD. MAGNETITE 20KV SPOT

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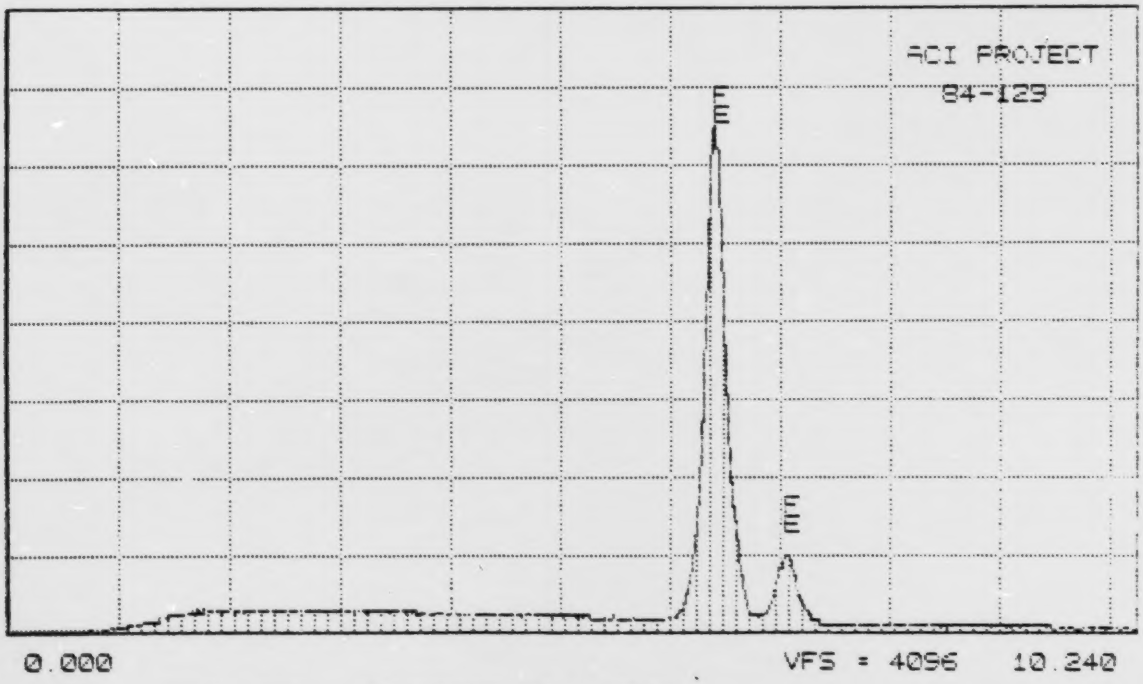

50   MIN STD. HAEMATITE 20KV SPOT

4

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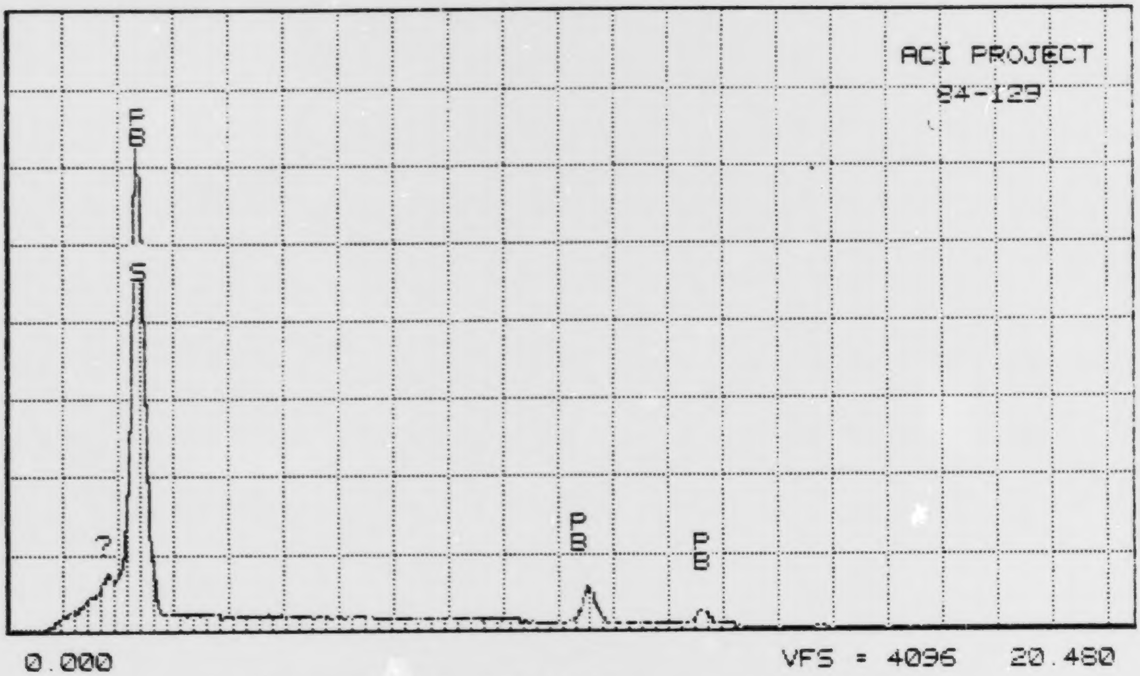

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50 MIN STD. GALENA 20KV SPOT

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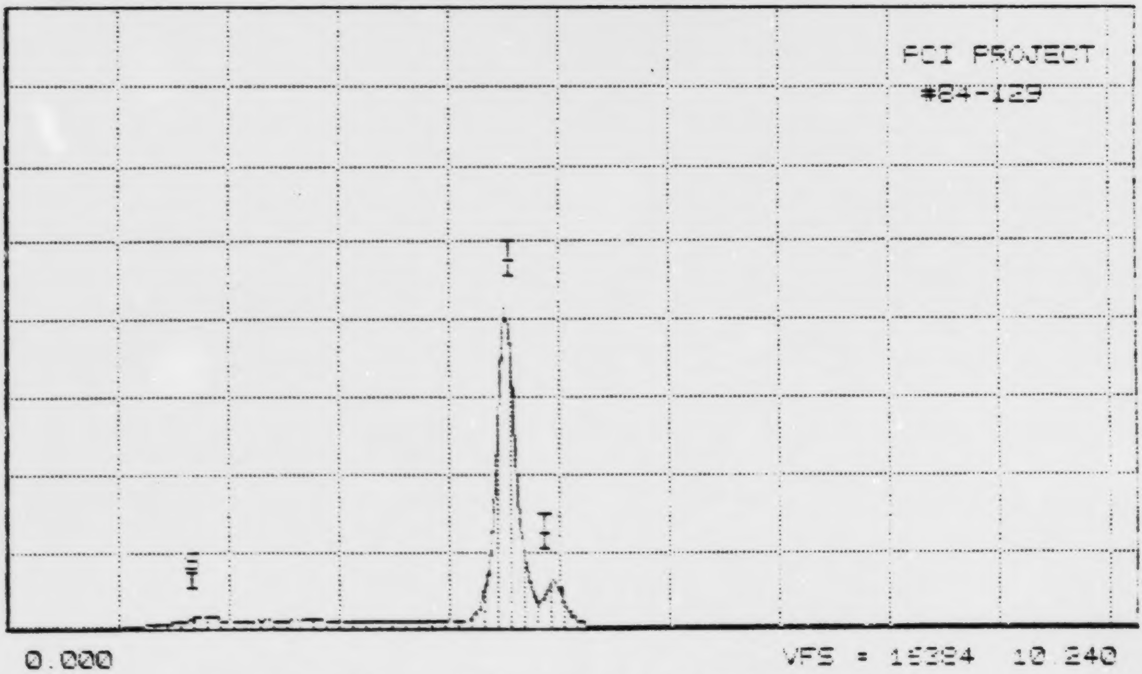

6

50 DUST MINERAL STANDARD RUTILE 20KV SPOT

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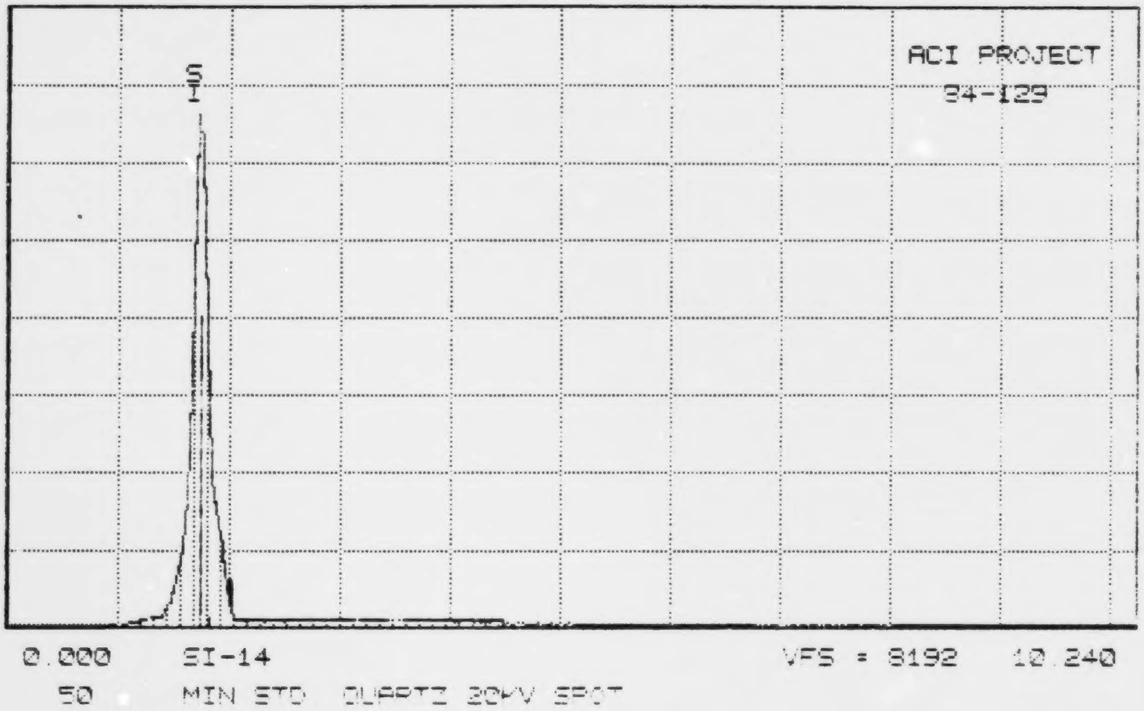

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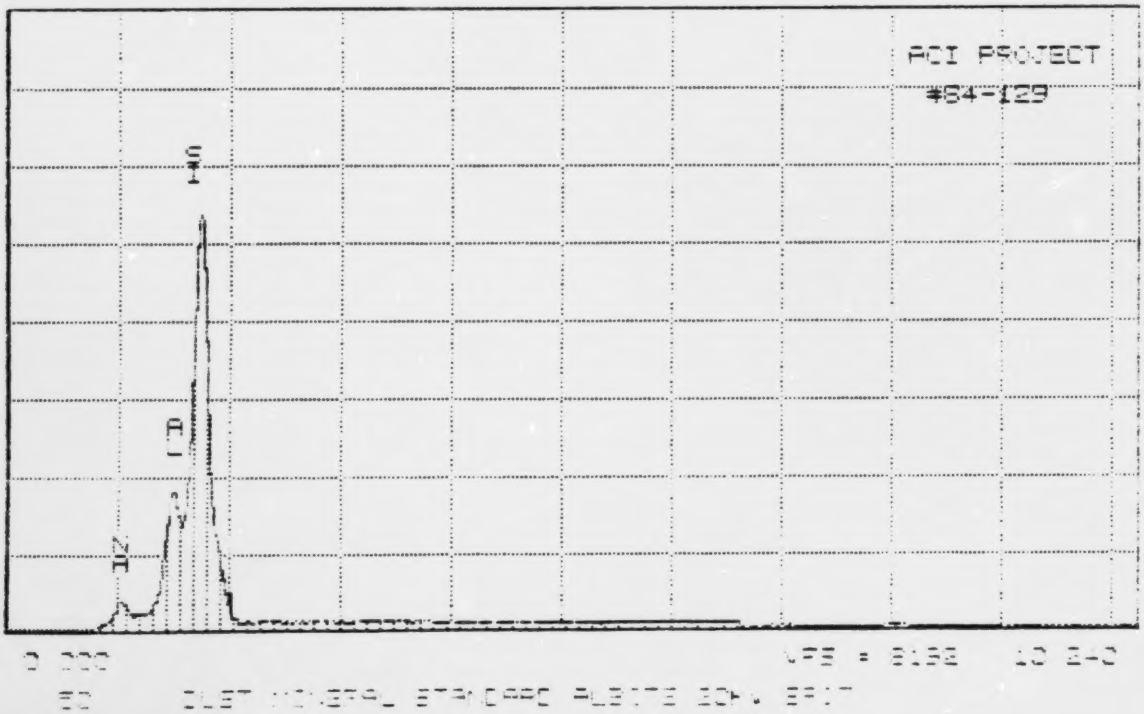

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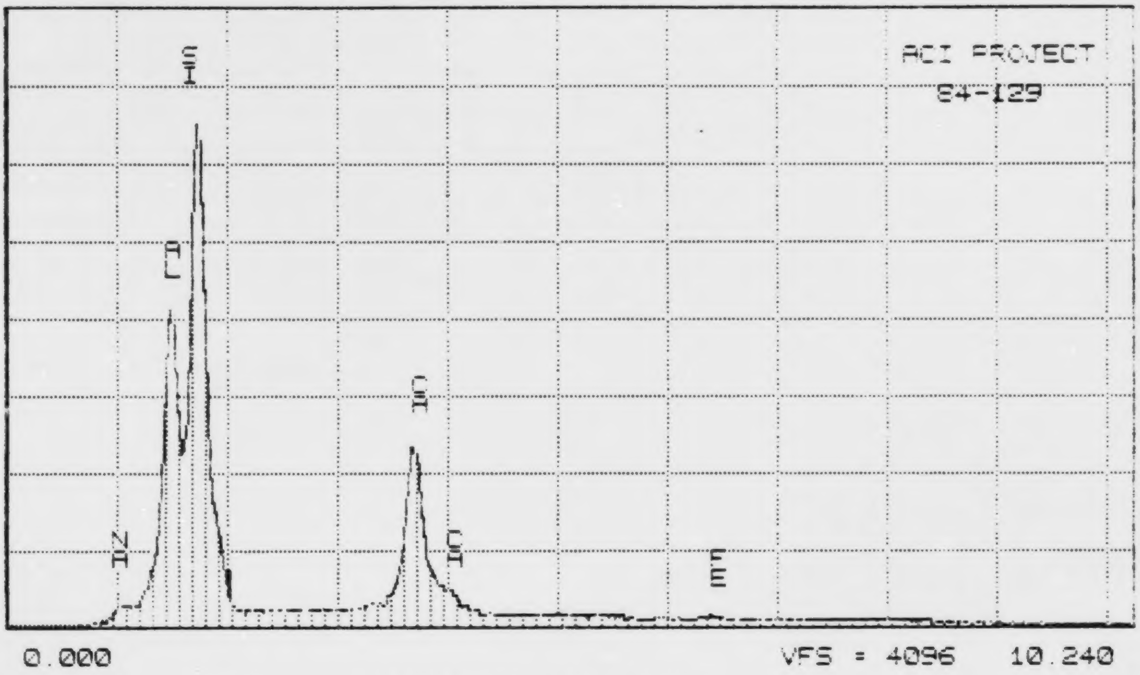

50 MIN STD. PLAGIOCLASE 20KV SPOT

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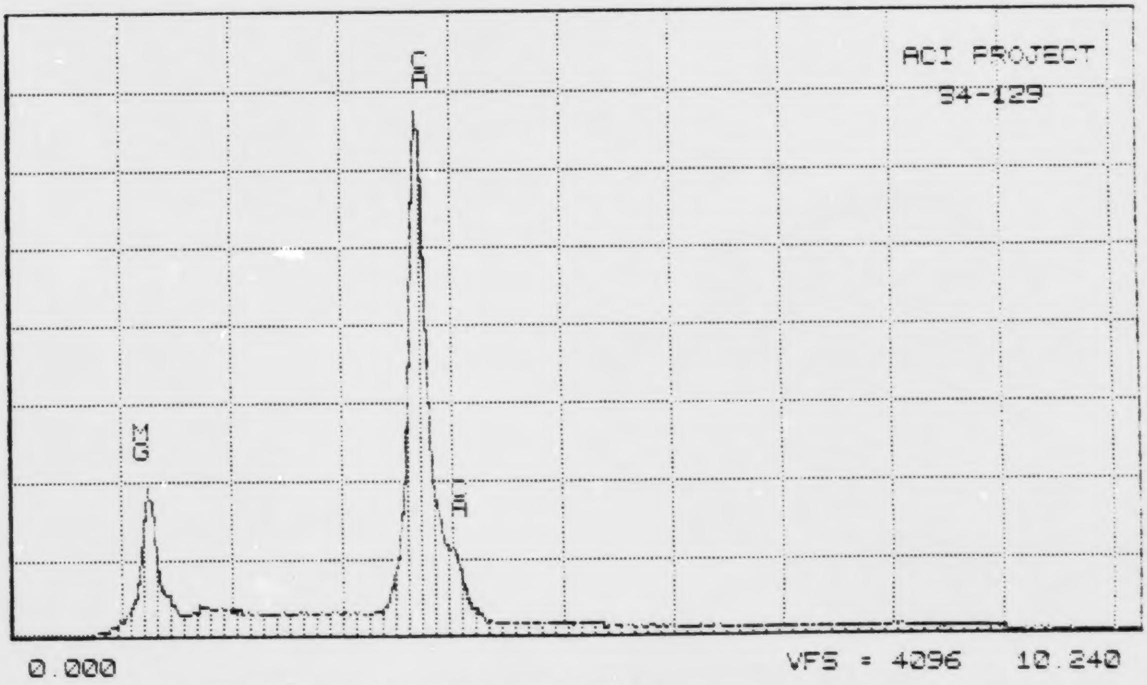

50 MIN STD. DOLOMITE 20KV SPOT

10

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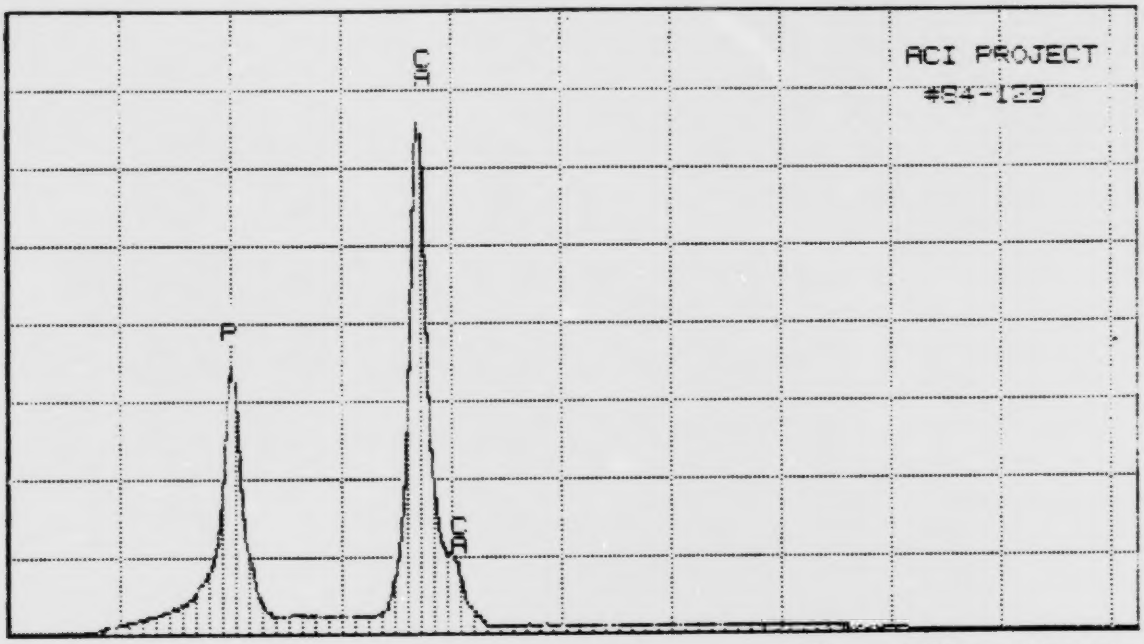

11

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50 DUST MINERAL STANDARD APATITE 20KV SPOT

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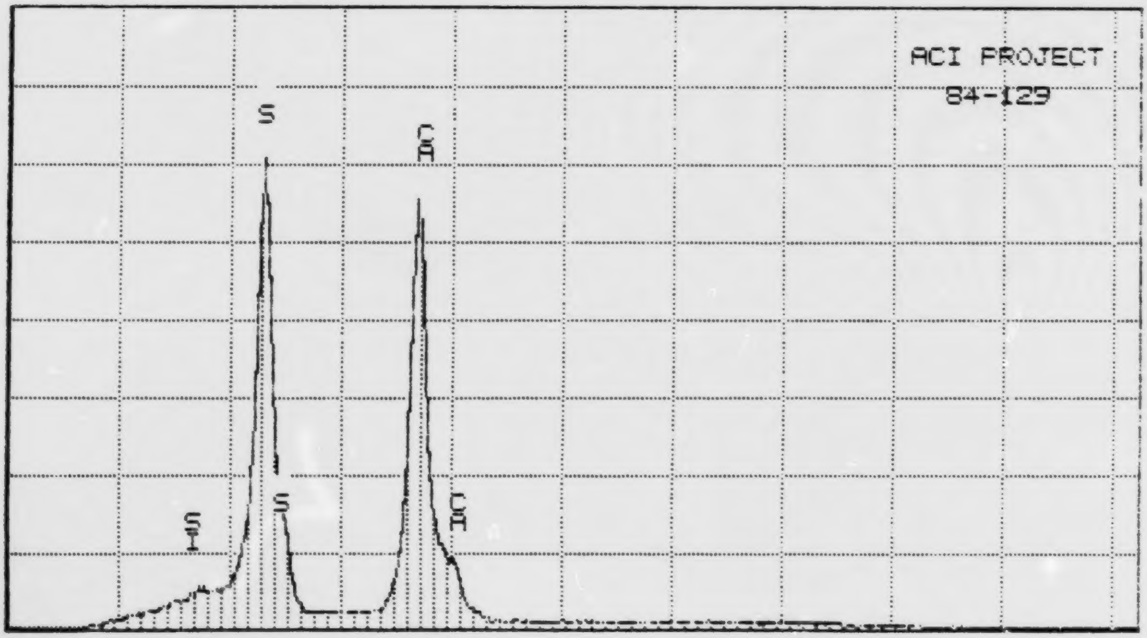

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50 MIN STD. CaSO4 20KV SPOT BEI

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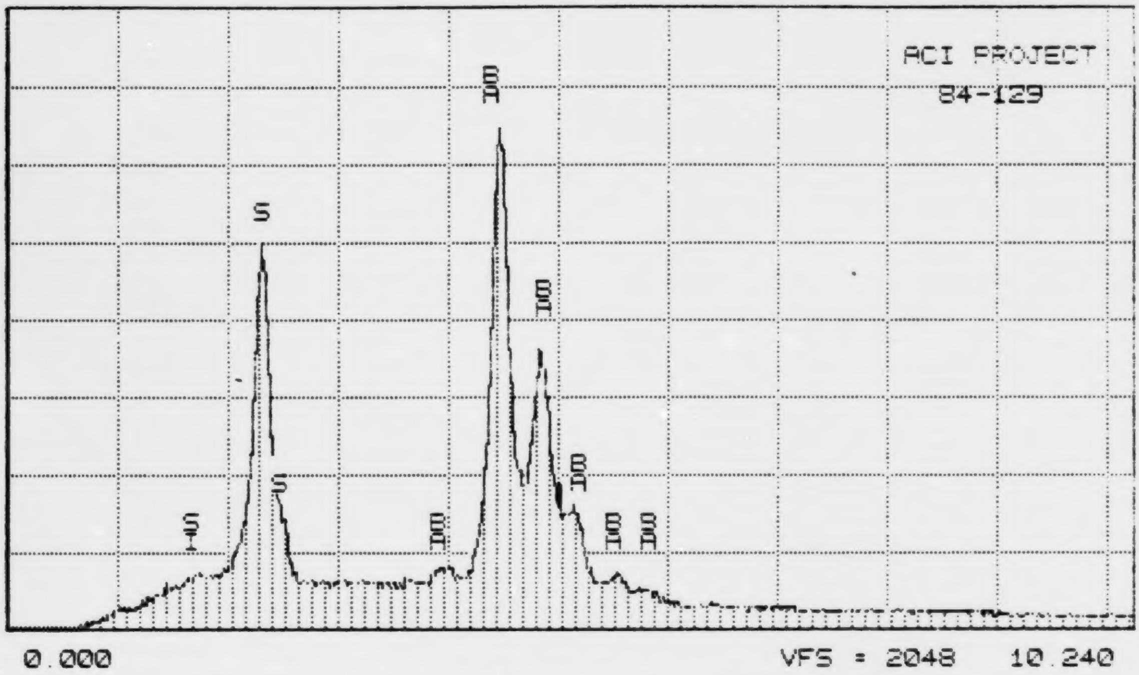

50 MIN STD. BaSO4 20KV SPOT BEI OUT

13

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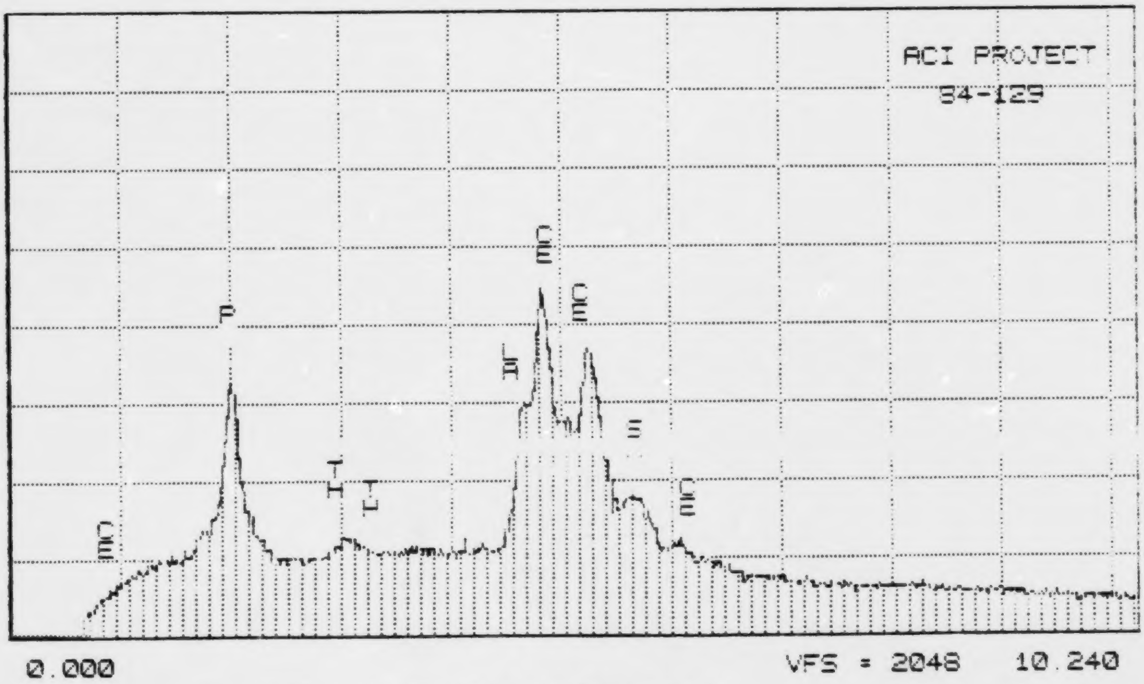

100 MIN STD. MONAZITE(2) 25KV SPOT

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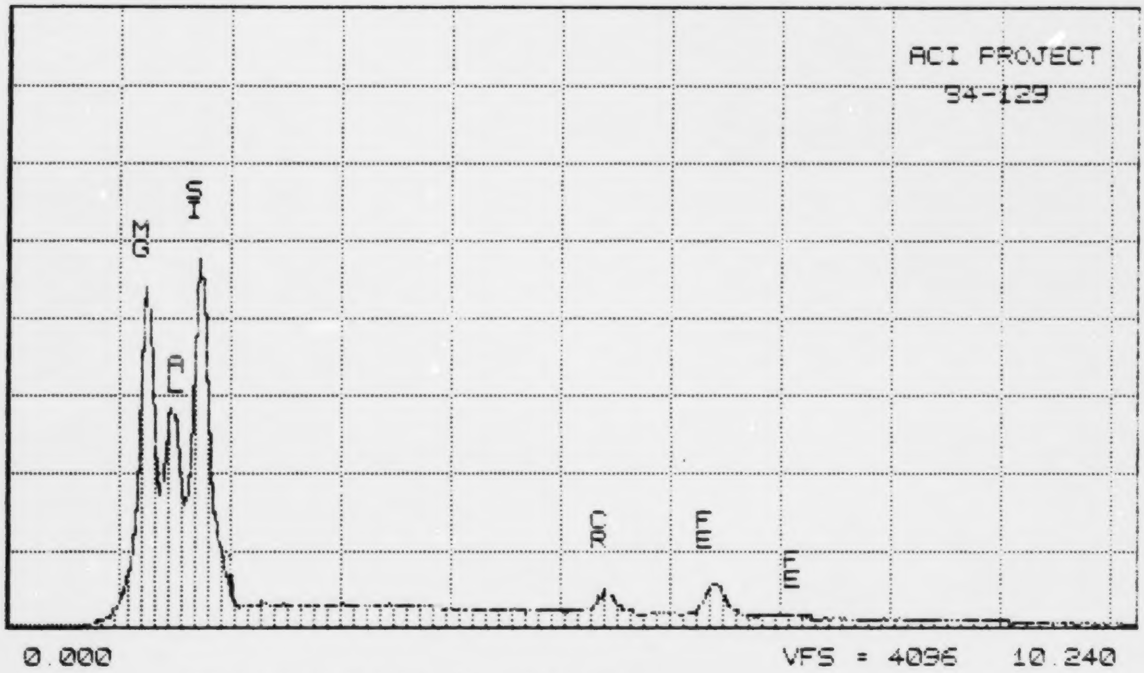

50 MIN STD. CHLORITE 20KV SPOT

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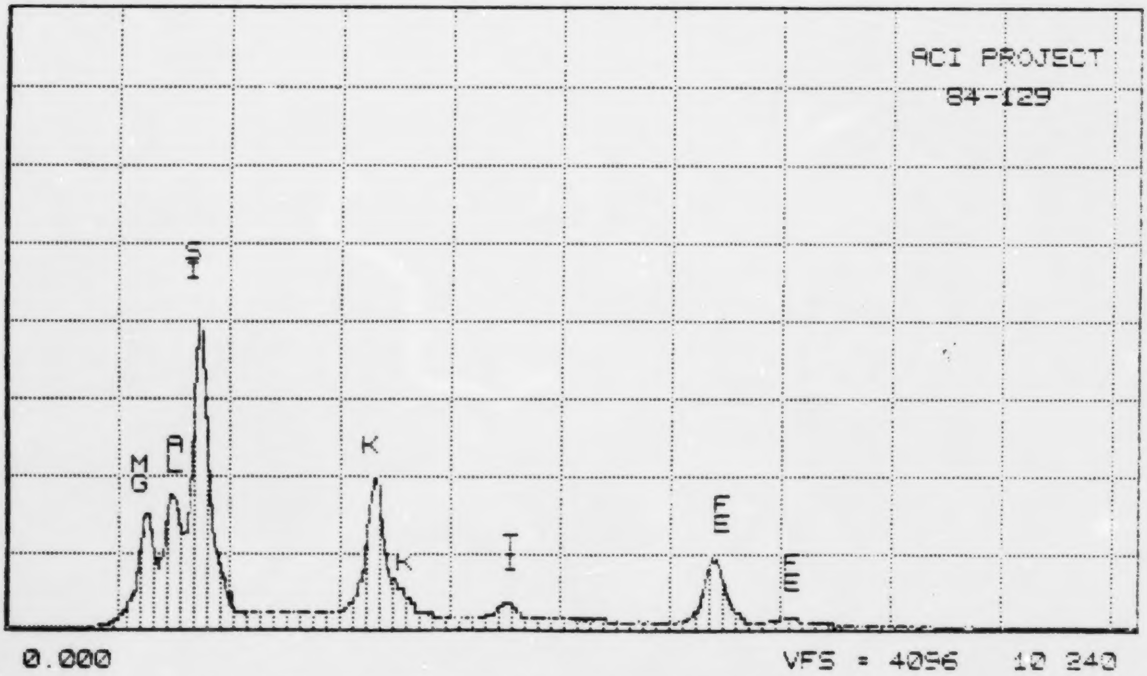
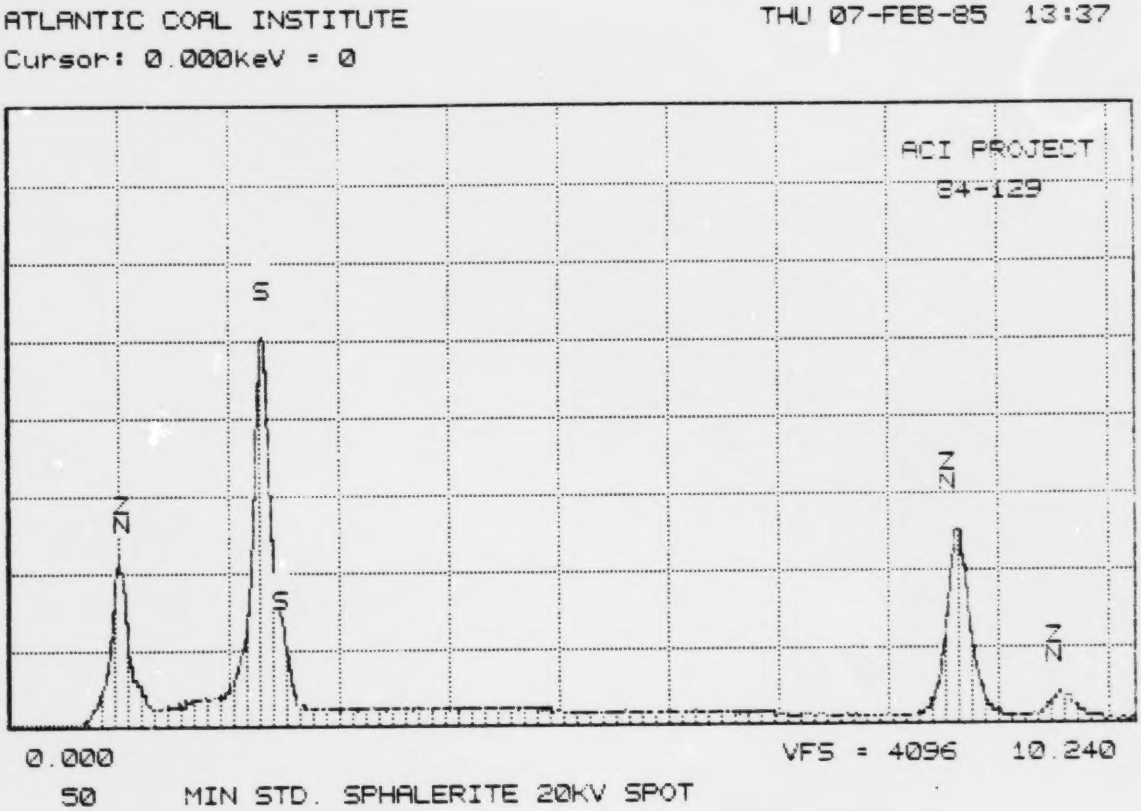


50 MIN STD. BIOTITE 20KV SPOT BEI OUT

16



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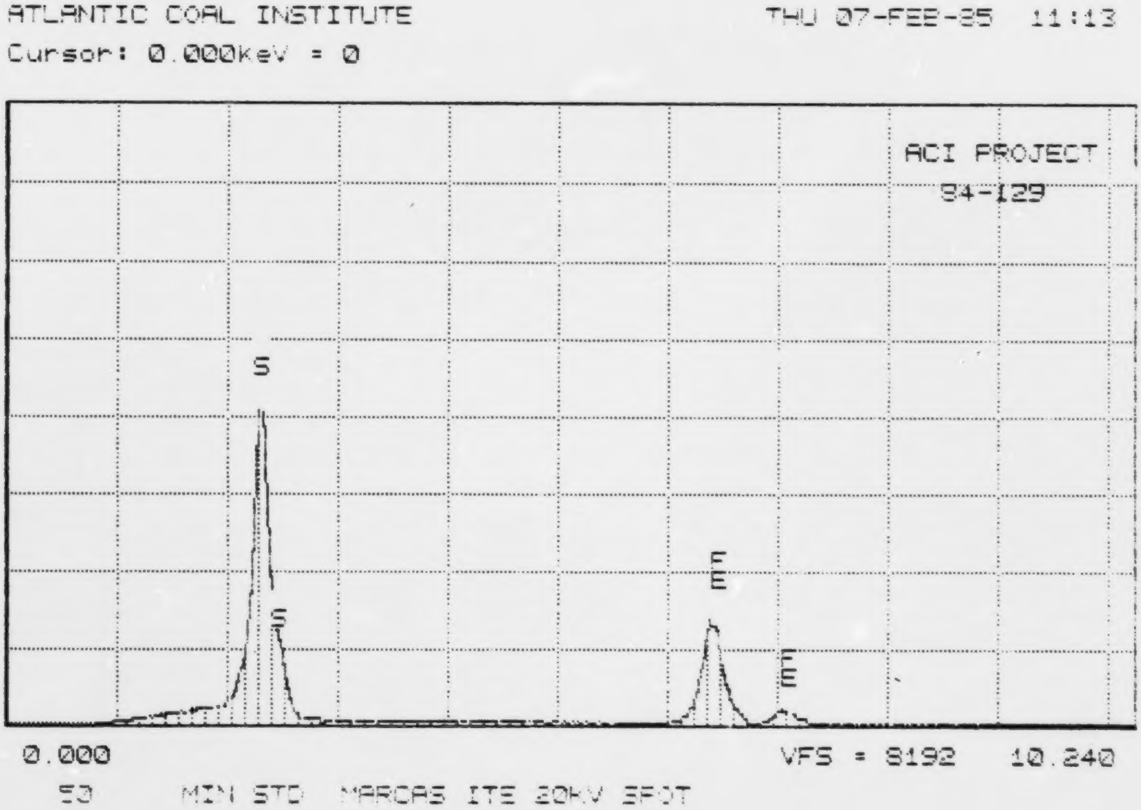
50 MIN STD. SPHALERITE 20KV SPOT

17



ATLANTIC COAL INSTITUTE THU 07-FEB-85 11:13

Cursor: 0.000keV = 0

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50 MIN STD MARCASITE 20KV SPOT

18

3a. PHOTOMICROGRAPHS: FILTER SUBSTRATES

1. Whatman Glass Microfibre filter, no rated pore size, gold coated, x1000
2. ibid. x7500
3. MSA Glass Microfibre filter, no rated pore size, gold coated, x1000
4. ibid. x7500
5. Nuclepore polycarbonate capillary pore membrane filter, 0.8um pore size, gold coated, x1000
6. ibid x7500
7. Silver metal membrane filter, 5.0um pore size, gold coated, x1000
8. ibid. x7500
9. MSI Micronsep nylon membrane filter, 0.45um pore size, gold coated, x1000
10. ibid x7500
11. Gelman Sciences vinyl metricel membrane filter, 5.0um pore size, gold coated, x1000
12. ibid x7500
13. Nuclepore membrafil cellulose ester membrane filter, 0.80um pore size, gold coated, x1000
14. ibid x7500
15. MSI Micronsep cellulose ester membrane filter, 0.80um pore size, gold coated, x1000
16. ibid x7500
17. MSA cellulose ester membrane filter, 0.80um pore size, gold coated, x1000
18. ibid x7500
19. Millipore cellulose ester membrane filter, 0.80um pore size, gold coated, x1000
20. ibid, x7500

21. MSA polyvinyl chloride (type FWS-B) membrane filter, 5.0um pore size, gold coated, x1000

22. ibid, x7500

23. Stainless steel stub surface polished and ultrasonically cleaned, x1000

24. Carbogel coal particles on polished aluminium stub, x1000

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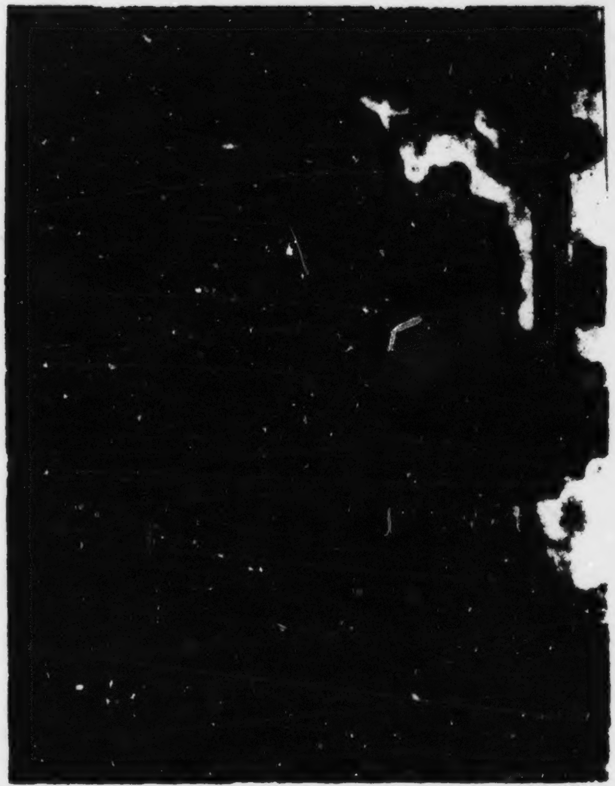

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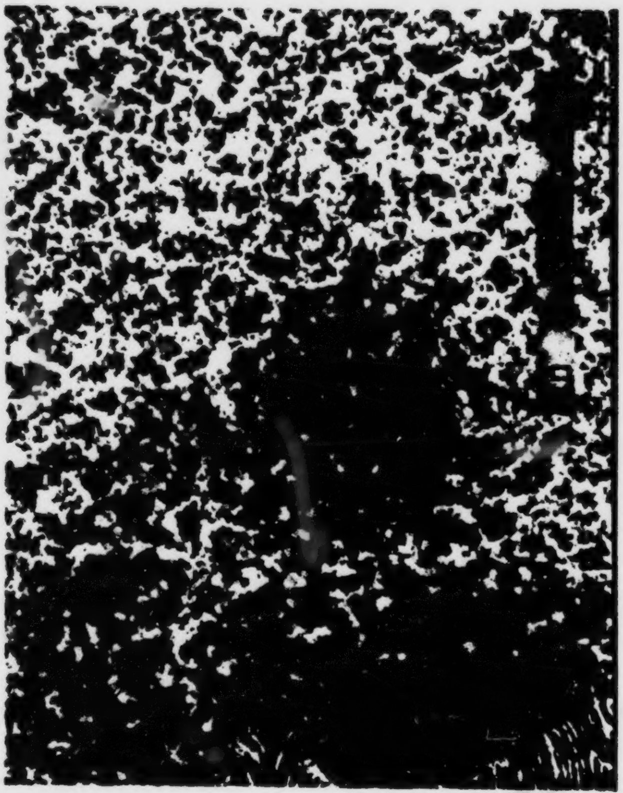

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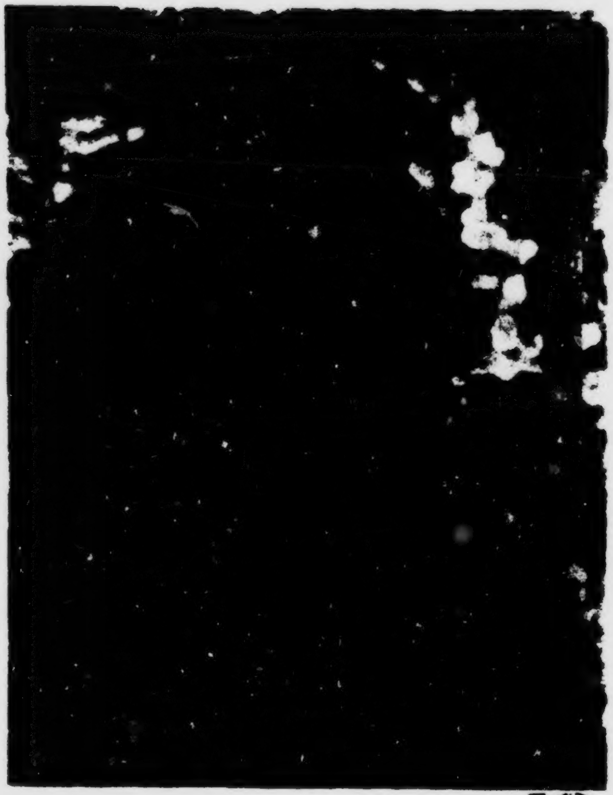

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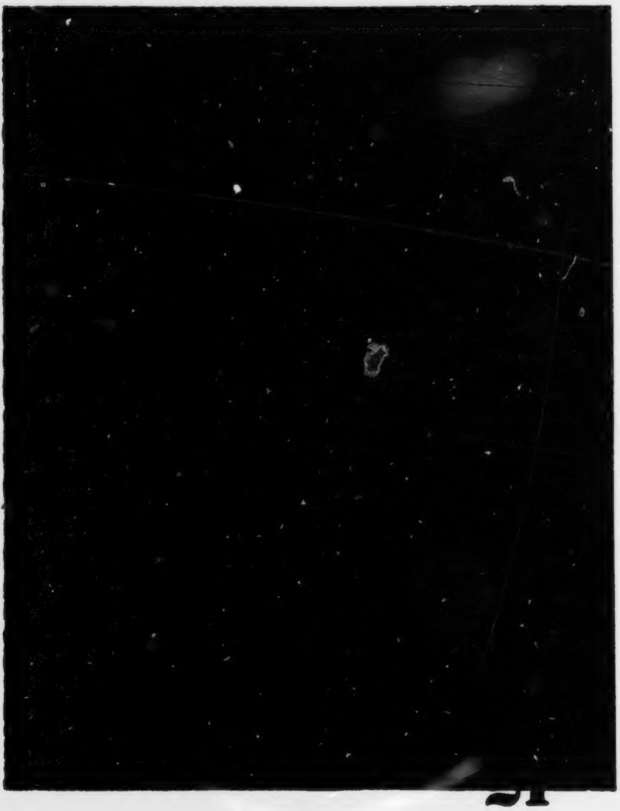

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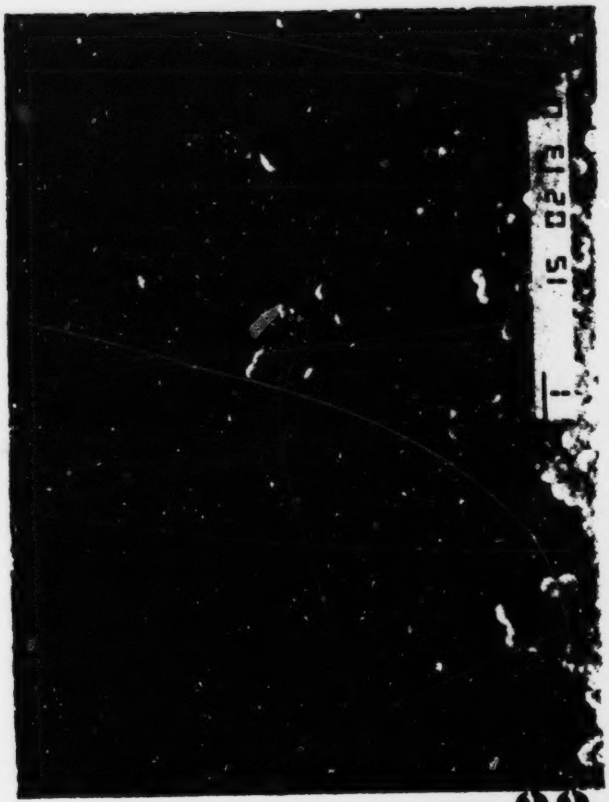

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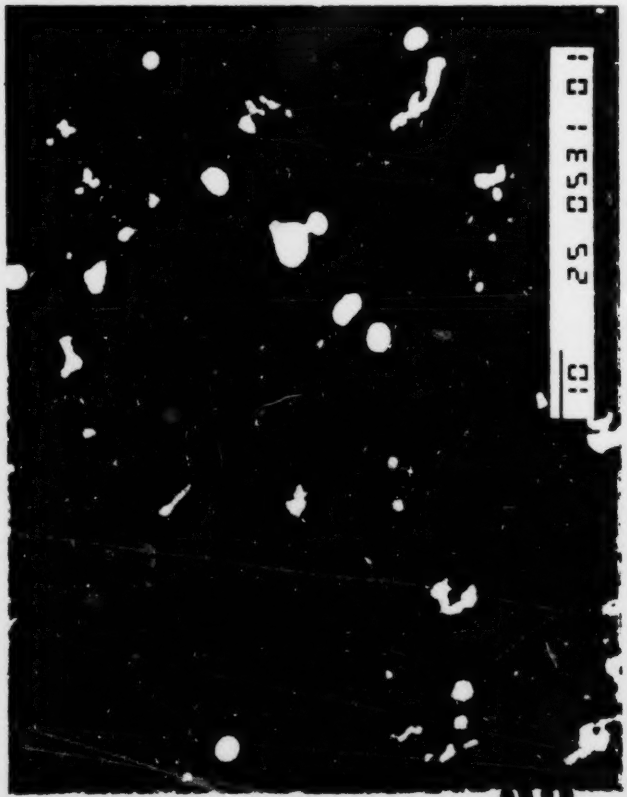
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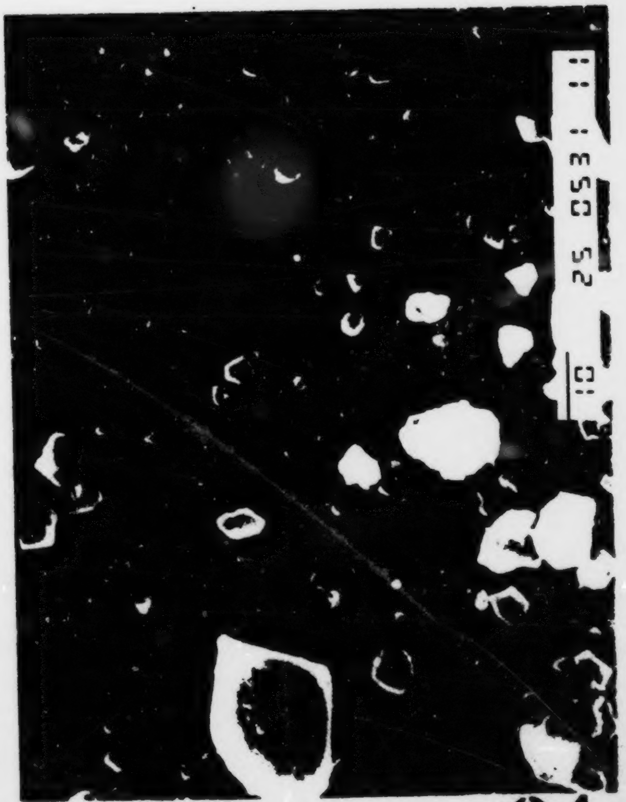
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24

3b. EDX SPECTRA: FILTER SUBSTRATES

1. EDX spectrum for an area raster on MSA Fiberglass at 20kv, 50 second count. The high count rates for Si result in a high vertical scale, suppressing the continuum background. The gold peak is a coating artifact but adds little to the complex spectrum. This filter material would be mistaken for plagioclase (see Appendix II 2e, EDX #9).

2. Nuclepore polycarbonate filter: EDX spot analysis for 50 seconds at 20kv. Note the large background with low Z element bias and distinct Si, Al peaks. A truncated peak is present at the lowest energy. The low count rates and large background would not be mistaken for a mineral particle but could be misidentified as a coal maceral.

3. EDX spectrum on silver metal membrane. The large silver peaks interfer with potassium or chlorine K lines.

4. EDX spectrum for Micronsep Nylon filter. The largest peak is coating gold, indicating a dominantly low atomic number chemistry. The spectrum is essentially the Bremsstrahlung xray background. Silica may be present or may be an instrument artifact.

5. Millipore cellulose ester membrane filter analysed by 50 second area raster at x100 magnification and 20kv. The gold peak is a coating artifact. Note large background and Si, Cu peaks. Al may also be present but has not been labeled. These peaks may also be due to instrument artifacts.

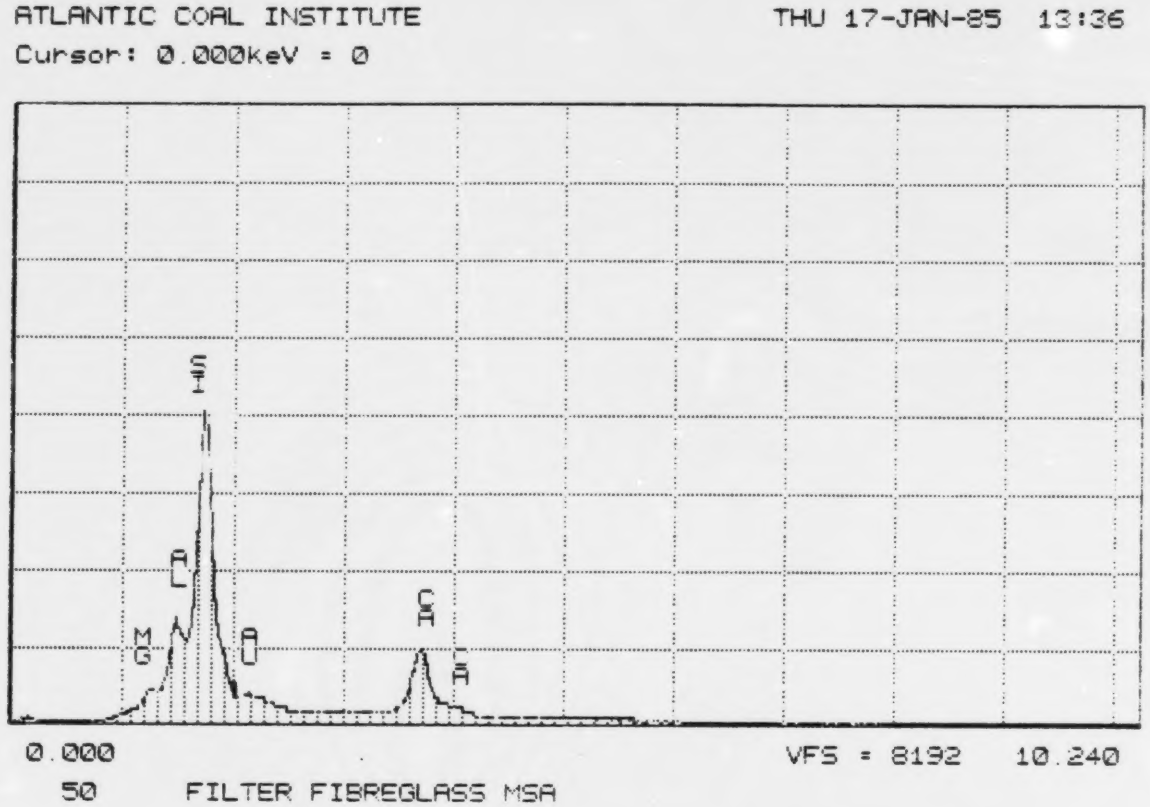
6. MSA cellulose membrane filter: The EDX spectrum is similar to the Millipore brand if the vertical full scales are matched. The main contribution is the xray continuum.

7. Micronsep cellulose membrane filter: The EDX spectrum correlates with the previous two brands. The Si peak is labeled but is less pronounced than for the Millipore cellulose and may in fact be only the xray continuum.

8. The EDX area raster on Nuclepore cellulose ester membrane filter material shows a similar spectrum to other cellulose filters but includes contaminant Fe and Cu. These peaks may be due to instrument artifacts although they are not present in all samples.

9. MSA polyvinyl chloride filter (FWS-B): Spot analysis. Note dominating chlorine peak as well as a low Z element peak at the detector threshold. Such a spectrum could be misidentified as halite (Welton 1984 p.183).

10. Gelman Sciences vinyl Metrical membrane filter showing an EDX raster spectrum similar to the MSA PVC (EDX #3) but with additional calcium and phosporus. Note the large vertical scale which suppresses the continuum background.


ATLANTIC COAL INSTITUTE THU 17-JAN-85 13:36

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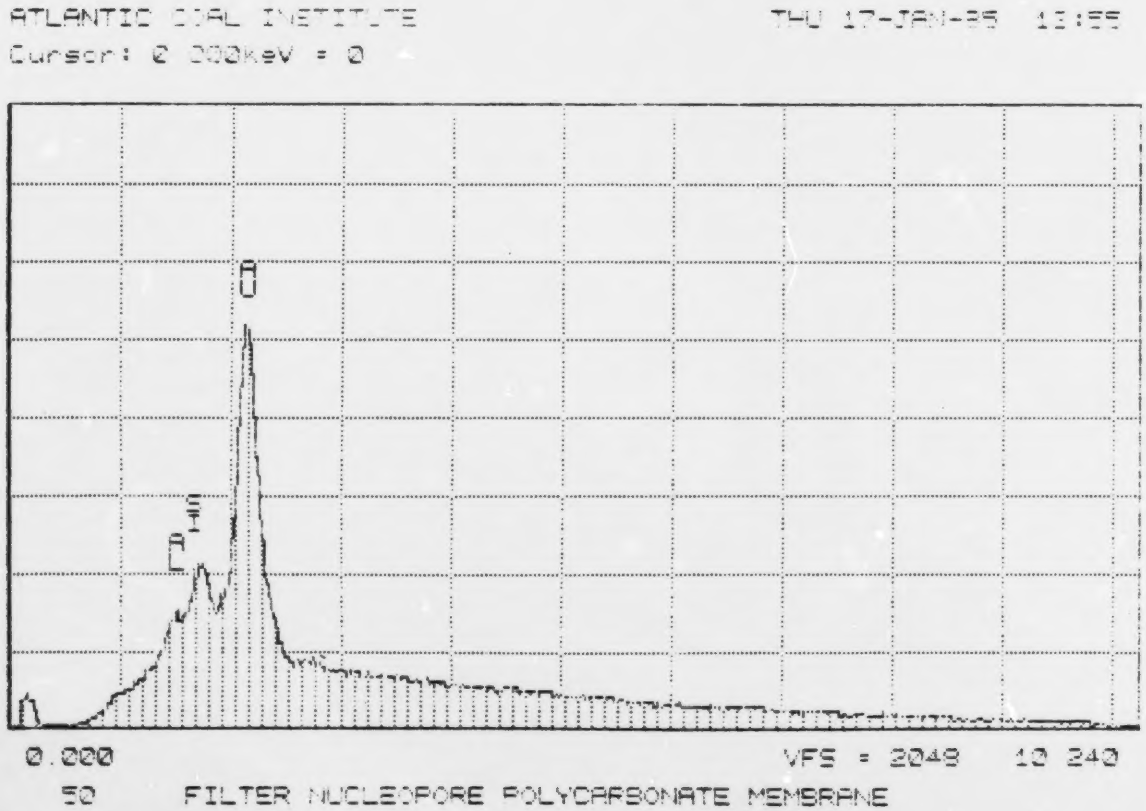
50 FILTER FIBREGLASS MSA

1


ATLANTIC COAL INSTITUTE THU 17-JAN-85 13:55

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50 FILTER NUCLEOPORE POLYCARBONATE MEMBRANE

2

ATLANTIC COAL INSTITUTE THU 17-JAN-85 13:42

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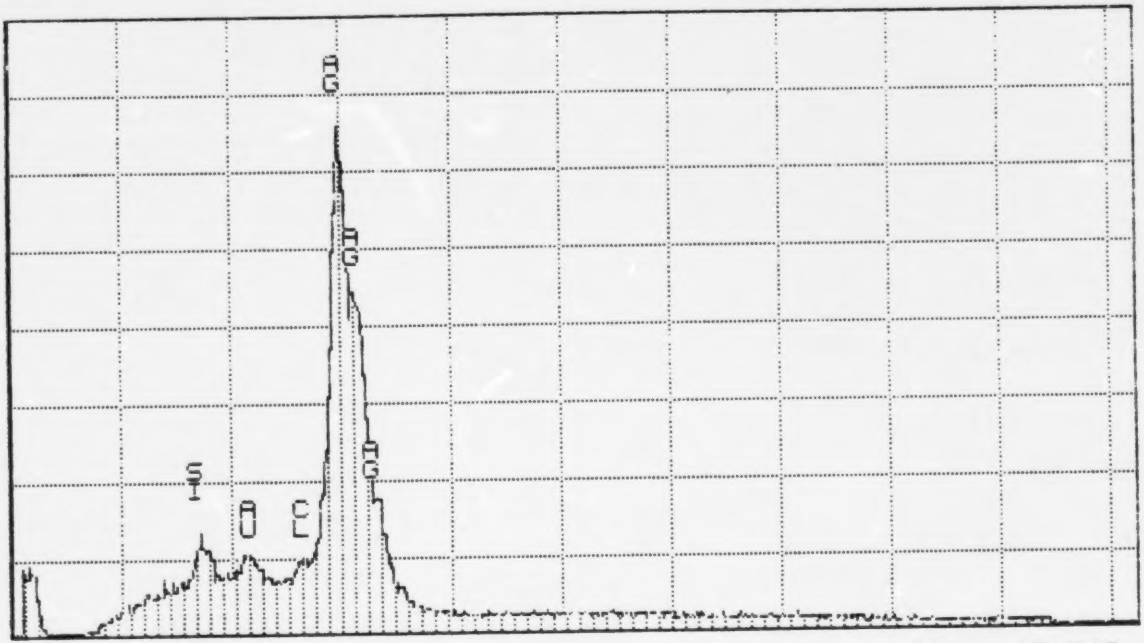

3

50 FILTER SILVER METAL MEMBRANE

ATLANTIC COAL INSTITUTE THU 17-JAN-85 13:48

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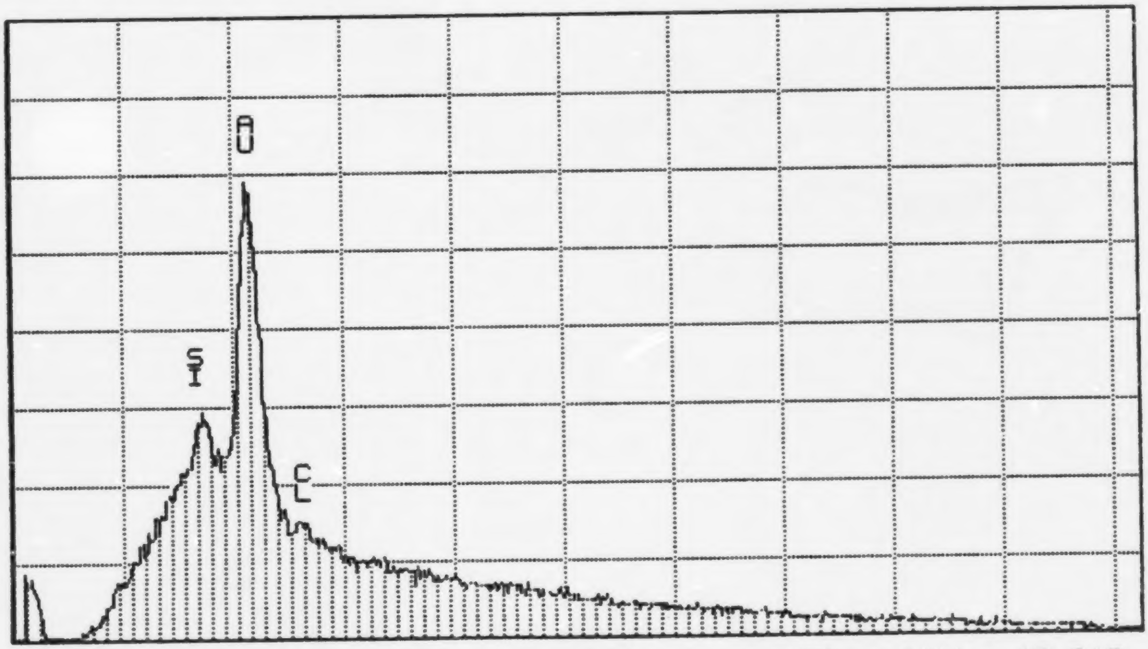

4

50 FILTER MICRONSEP NYLON (0.45)

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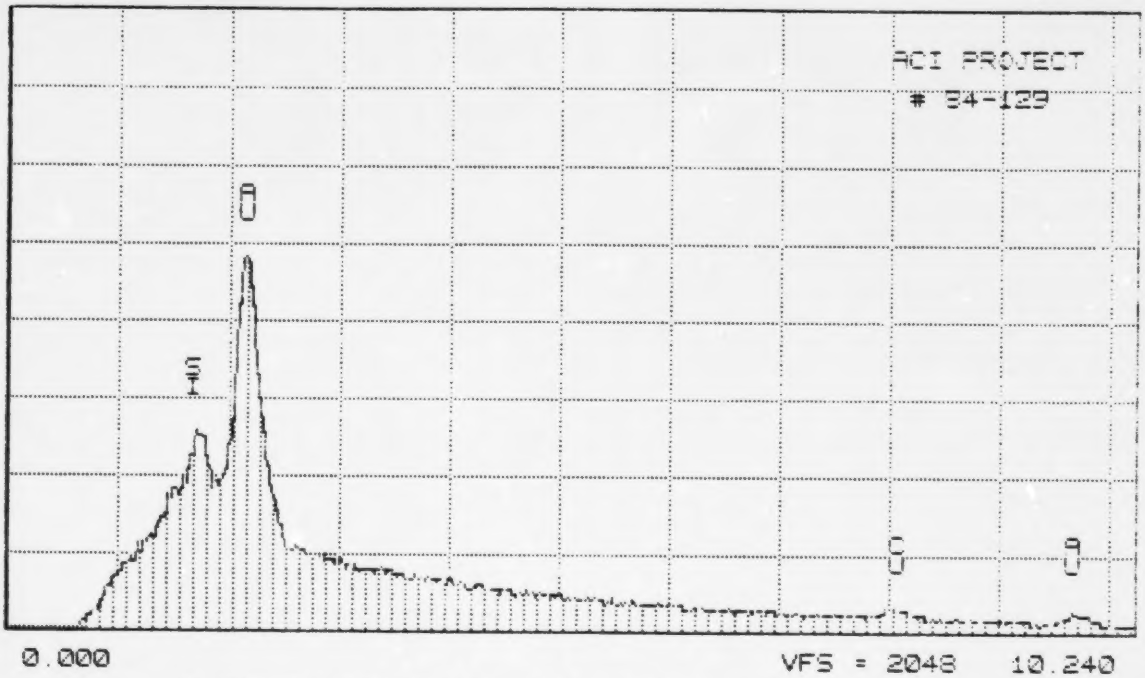

50 DUST MILLIPORE CELLULOSE FIL RAS-P1 100X SEI

5

ATLANTIC COAL INSTITUTE THU 17-JAN-85 13:38
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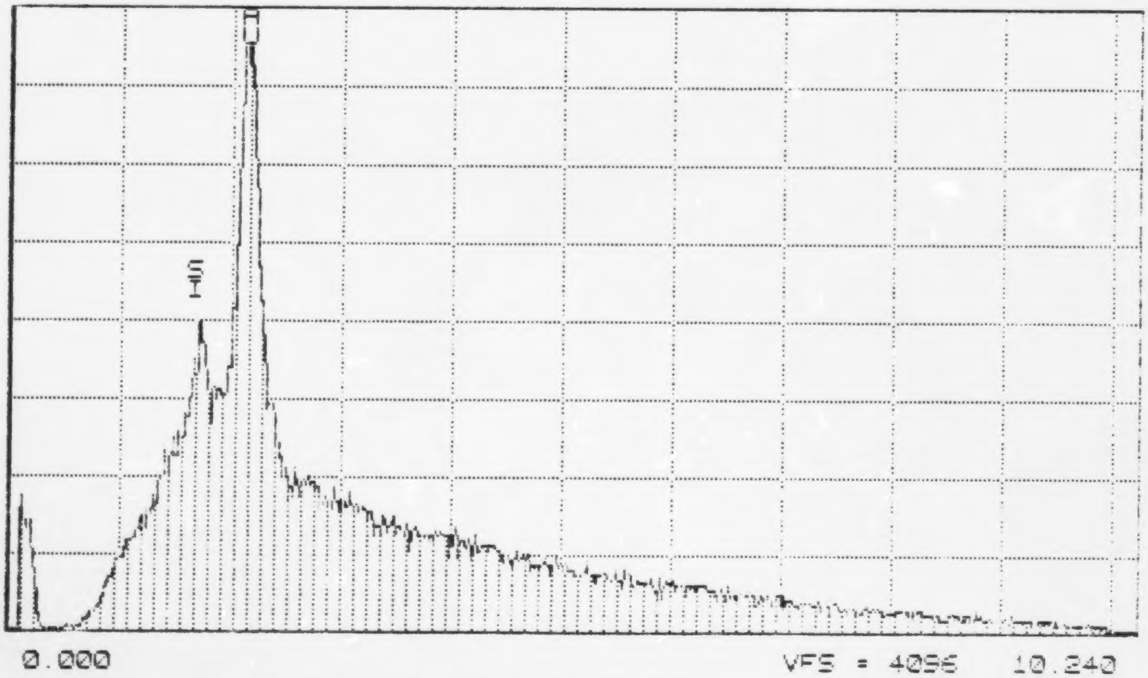
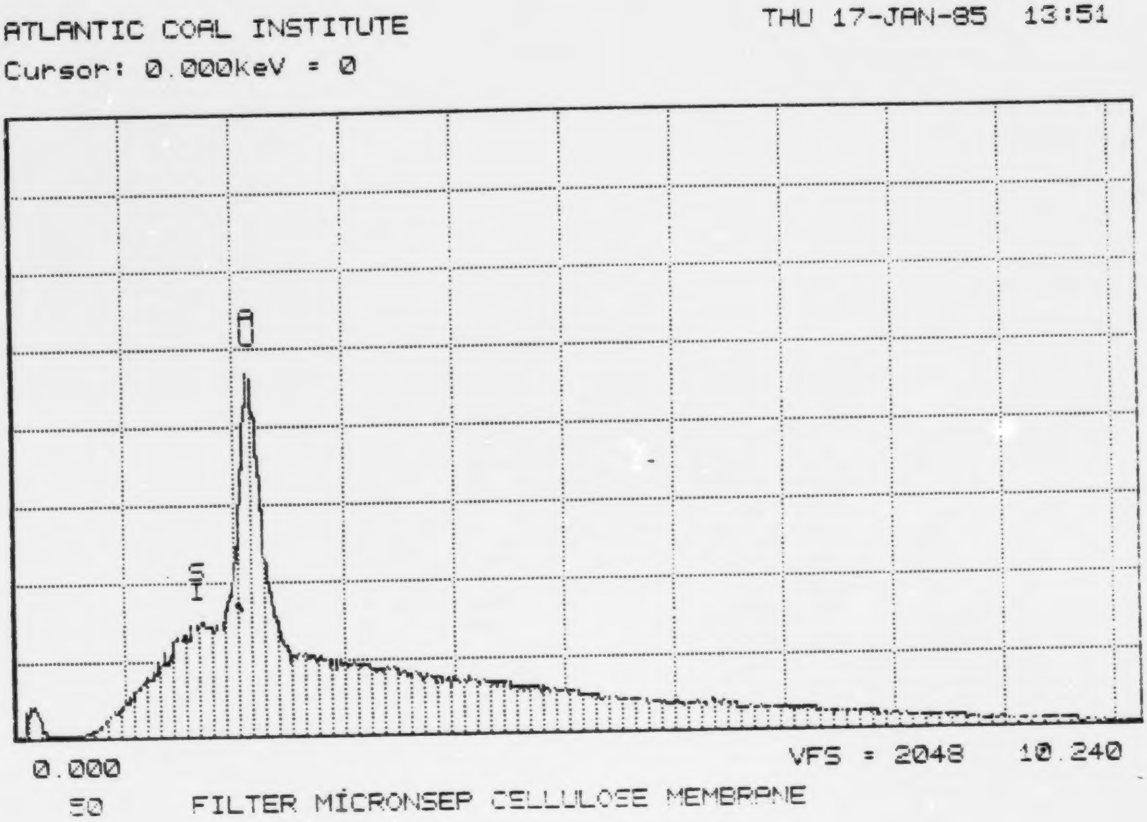


50 FILTER MSA CELLULOSE MEMBRANE

6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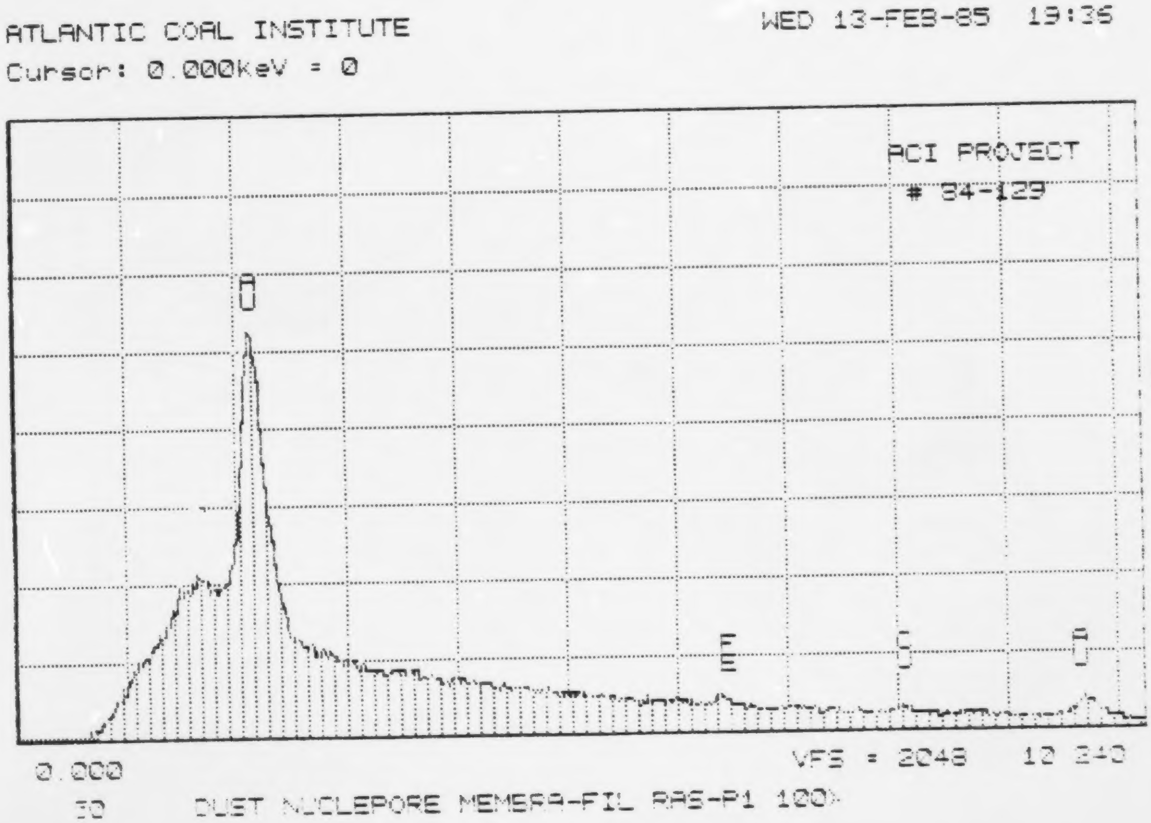
50 FILTER MICRONSEP CELLULOSE MEMBRANE

7


ATLANTIC COAL INSTITUTE WED 13-FEB-85 19:36

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ACI PROJECT

84-129

0.000 VFS = 2048 10.240

50 DUST NUCLEPORE MEMBRA-FIL RAS-P1 100>

8

ATLANTIC COAL INSTITUTE
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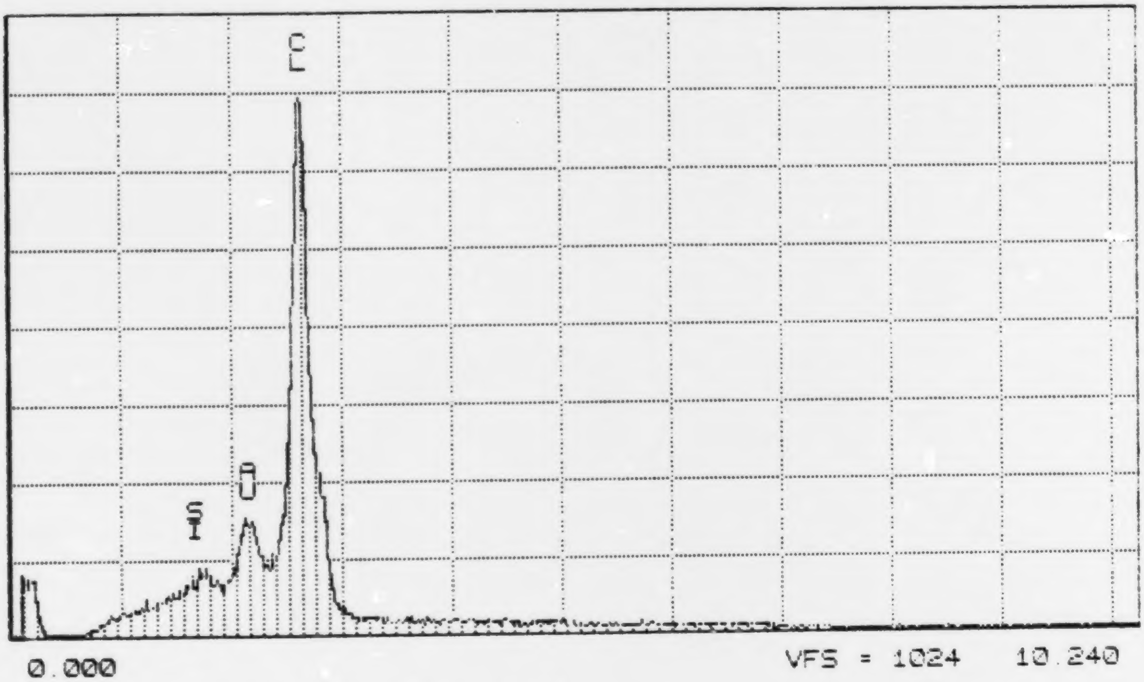

50 FILTER MSA FWS-B (PVC)

9

ATLANTIC COAL INSTITUTE
WED 13-FEB-85 21:13
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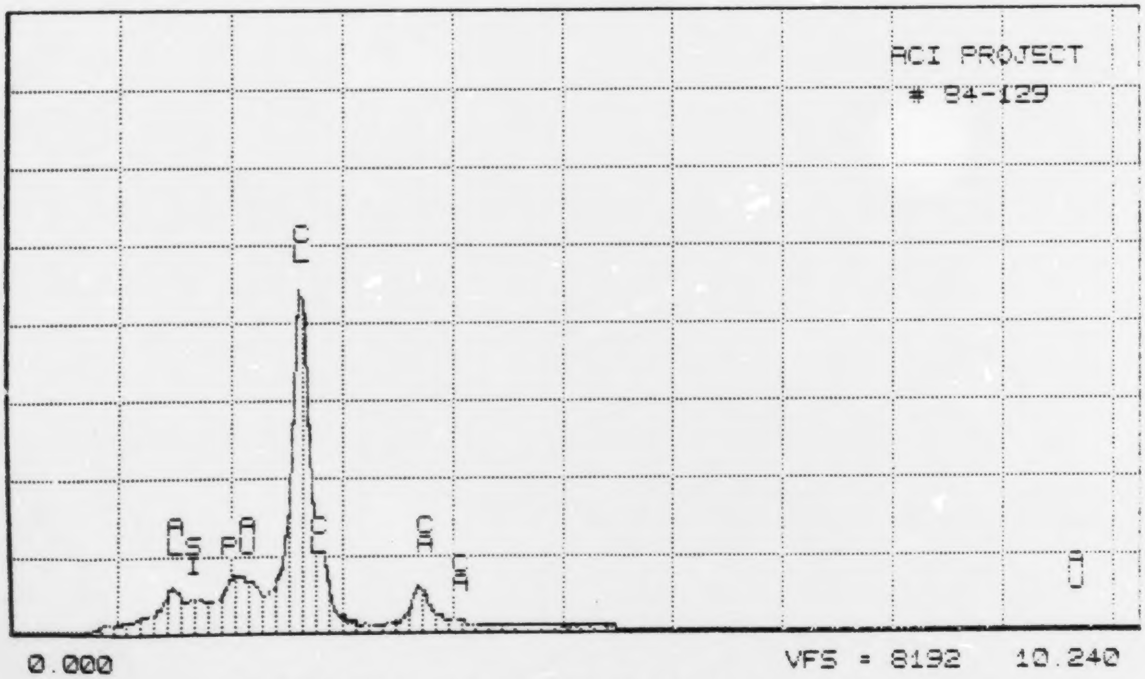

ACI PROJECT
84-129

50 DUST GELMAN VINYL METRICEL RAS-F1 100X

10

3c. PHOTOMICROGRAPHS: FILTER LOADINGS

1. Nuclepore polycarbonate filter (0.8 um pores) showing heavy loading of coal mine dust. The filter shows uneven loadings forming concentric concentration rings. This specimen has been sliced at the filter centre (top of photo), and gold coated. x35, SEI, 20kv.

2. As above but outer edge of filter, light loaded. Note the particle morphologies and size distribution. Coals are generally larger angular fragments or elongate slivers. In SEI mode, the grey contrasts are due largely to charging effects plus some backscattered electron contribution reflecting mean atomic number. x1500, SEI, 20kv, gold coated.

3. Very lightly loaded cellulose ester type filter. Gold coating, x200, BEI, 15kv.

4. Enlargement of photo 3 field showing the poor contrast of small dust particles against the filter matrix. Such a light loading is not suitable for investigations because of the limited number of particles at even moderate magnification. SEI, x1500, 15kv.

5. Millipore cellulose ester filter with moderate loading of coal dust. Note the streaked image due to charging. This sample is carbon coated which is less effective than gold for such rough topography. x750, SEI, 20kv.

6. Enlargement of field from photo #5. Note the poor quality image due to charging effects and the poor contrast of particles to filter substrate. x7500, 20kv.

7. PVC filter loaded with coal dust. Note the uneven distribution controled by the pore configuration. Au coated, x2000, SEI, 15kv.

8. Enlarged field on loaded PVC filter taken in backscattered electron image mode. Note the particle grey shades reflecting different mean atomic number. Particles are bunched in the filter matrix making individual particle analysis difficult. BEI, x500, 20kv.

9. Nuclepore polycarbonate filter (8um pores) with light loading of coal dust. Note pore diameter compared to photo 2, and the charging at pore edges. Grains show bright edges due to beam charging. x1000, Au coated for 60 seconds, SEI, 15kv.

10. Same field of view as photo 9 but in backscattered electron mode. Note that bright grains in photo 9, due to charging, are now low contrast due to low mean atomic number chemistry. These are likely coal dust. Note that charging at pore edges is absent for BEI mode, but the image is less sharp.
    Bright grains under BEI are coal minerals, stone dust, or roof rock contribution with higher atomic number elements present. x1000, Au coated, 15kv.

11. Nuclepore polycarbonate filter (0.8um pores) with moderate coal dust load, gold coated (120 seconds). Note pores still show charging, but particles are grounded compared to photo 9. This is due to the longer coating time. x1500, SEI, 15kv.

12. Same field as photo 11 but in BEI mode. Pores show no charging, but image is less sharp. Particle grey contrasts are weak at 15kv and could be enhanced if conditions upgraded.

13. Dust particle cluster on fibre glass filter viewed at high magnification in SEI mode. Note the poor resolution between particles. x10,000, Au coated, 15kv.

14. Same field of view as photo 13 but in BEI mode. Note the marked improvement in particulate resolution. The mottled bright grain has a higher mean Z than adjacent grains, but heterogeneous chemistry. x10,000, BEI, 15kv.

15. LTA composite redeposited on Nuclepore polycarbonate filter and gold coated. Note the poor particle differentiation in SEI mode except for the bright charged grain. x5000, SEI, 20kv.

16. LTA composite: same field as photo 15 but in BEI mode and x3500. The bright grain is easily differentiated from the lower mean Z material. A matching EDX spectrum is given in Appendix , EDX #5, and shows a copper sulphate or sulphide of unknown mineralogy. x3500, BEI, 20kv.

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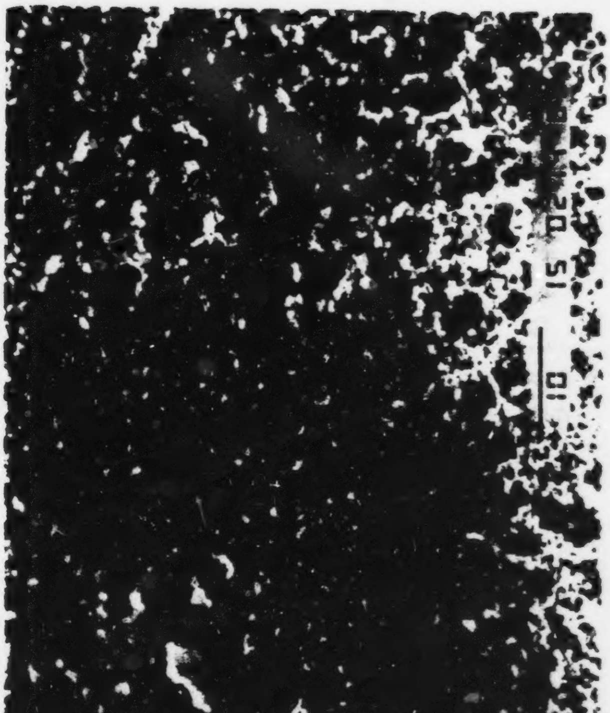

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4a. PHOTOMICROGRAPHS: LOW TEMPERATURE ASH

1. Low temperature ash from a composite of shearer operator respirable dust filter samples (85-2, 85-34, 85-72,Appendix 1 Table 4). The dust was ashed for 15 hours and redeposited on a Nuclepore (0.2um pore) filter. EDX spectra (#1-6) show the presence of macerals (1), antlerite (?) Cu3(OH)4SO4 (spot 2), chlorite (3), kaolinite (4), pyrite (5), and oxidized pyrite (6). See EDX #1-6. x750, gold coated, 20 kv. BEI.

2. ibid. x1000. Minerals present include kaolinite (1) illite (2-5). Note the charging effects creating bright areas. SEI, 20kv., see EDX #7-12.

3. ibid. x500. Minerals present include pyrite (1) illite (2), siderite? (3). BEI. see EDX #14-17.

4. ibid. x1000. Calcite cleavage fragment. see EDX #20.

5. ibid. x3500, SEI, pyrite (FeS2) (EDX #18)

6. ibid. x1500 SEI chalcocite (?) (CuS) (EDX #19). Mineral identification is tentative.

7. ibid. x3500 SEI pyrite (EDX #13)

8. ibid. x5000 SEI pyrite (EDX #21)

9. ibid. x5000 SEI. Pyrite framboid. Compare with LTA coal ash sample. Appendix II 1a, photo 6.

10. Respirable coal dust sample #85-9 collected on Millipore (0.8 um pores) and low temperature ashed on a gold coated glass slide. x35, SEI, 20kv.

11. Enlargement of photo #10, showing flocculated nature of the ash and particle agglomeration. x5000 SEI.

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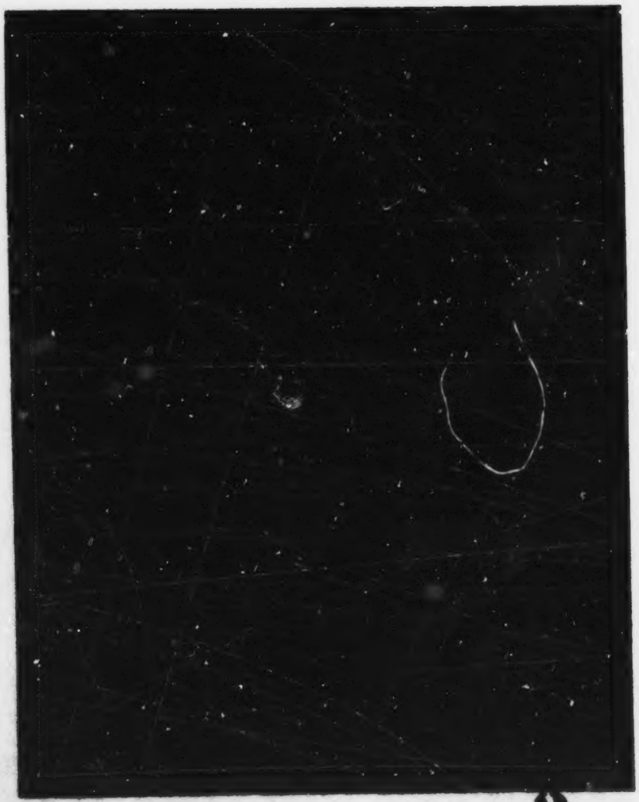

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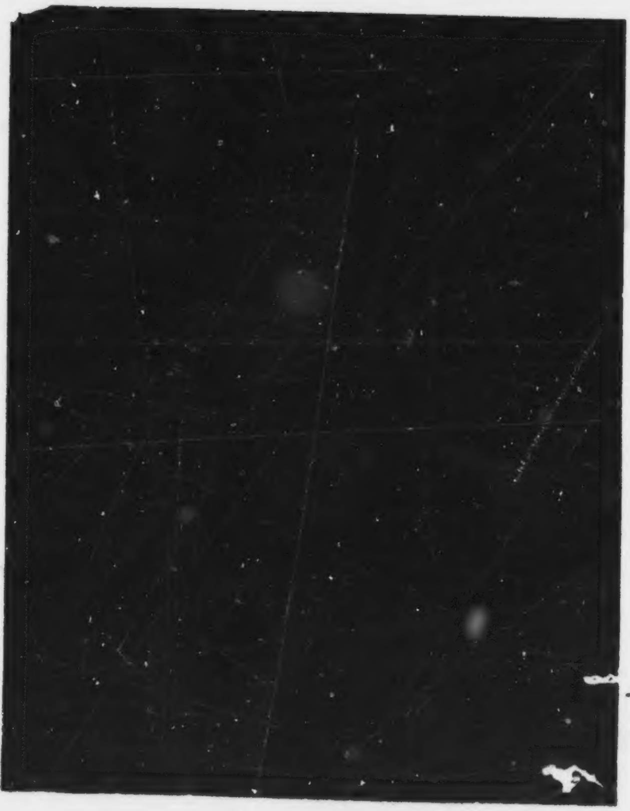

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4b. EDX SPECTRA: LOW TEMPERATURE ASH

1. LTA Comp sample: low temperature ash from a composite of shearer operator samples (85-2, 85-34, 85-72) redeposited on Nuclepore (0.2 um pores). This EDX spectrum is based on a spot analysis at 20 kv for 10 seconds. Note the very low counts (VFS) and exaggerated background, indicative of an organic particle (Spot #1, photo #1). The gold is coating artifact while the Cu, Fe, and Si peaks may be partially instrumental.

2. ibid spot #2. A copper rich mineral not previously reported for Sydney coalfield samples. Tentative identification is Antlerite (?) Cu3(OH)4.SO4 based on a Cu:S ratio of 3:1.

3. ibid spot #3. chlorite (?) $Mg3(Si_4O_{10})(OH)_2\ Mg_3(OH)_6$

   Note the absence of Al compared to our Astimex chlorite standard (Appendix II 2e, EDX #15). The chemistry and fibrous morphology is also compatible with other mineral identifications such as serpentine (chrysotile variety), anthophyllite or sepiolite. This illustrates the limits of SEM-EDX identification without additional information.

4. ibid spot #4. Kaolinite $(Al_4Si_4O_{10})\ (OH)_8$.

5. ibid spot #5. Pyrite (FeS2).
   The additional peaks of Al, Si, and trace K, Ca are interpreted as spurious due to stray xrays.

6. ibid spot #6. Pyrite oxidized by the LTA process. The presence of a Ca peak is interpretated as due to fine stone dust adhered to the pyrite grain or stray xrays from adjacent stone dust grains.

7. ibid photo 2. Spot #1. Kaolinite.

8. ibid spot #2. This spectrum may be a composite. The angular particle shape (photo 2) suggests a coal maceral which would account for the large background. The chemistry likely includes contribution by finely disseminated illite and pyrite.

9. ibid spot #3. This spectrum is likely for illite: $K(Al,Fe)_4(Si, Al)_8O_{20}\ (OH)_4$.

10. ibid spot #4, illite. Note the attenuated K peak, either due to particle geometry or chemistry.

11. ibid spot #5. This is a composite spectrum, either of carbominerite (coal, illite, pyrite, etc. interbedded) or of multiple particles and spurious peaks.

12. ibid spot #6. This particle shows charging (bright edges) which may account partially for the complex spectrum. A conglomerate of clay, pyrite, stone dust, and a copper sulphate mineral (Antlerite?) is indicated.

13. ibid photo #7. Pyrite crystal colony showing typical FeS2 spectrum.

14. ibid photo #3. Pyrite framboid yielding typical FeS2 spectrum with geometric enhancement of the S peak due to the rough sample surface.

15. ibid spot #2. Likely an illite spectrum but compared to EDX #9, the Al peak is suppressed. This may be a geometry effect.

16. ibid spot #3. Siderite ? (FeCO3) or iron oxide (Fe3O4) from pyrite oxidation. All peaks besides Fe are considered spurious due to scatter from adjacent grains.

17. EDX spectrum from a coal maceral particle (spot #4, photo #3). Note the low counts shown by the vertical full scale (VFS). Si, S, and minor Al, Fe, Ca, K, and Ti are typical for Sydney coals. These elements are either locked in the organic matrix or occur as submicron size minerals.

18. EDX spectrum from the LTA composite sample: massive pyrite (photo #5).

19. ibid photo #6. A distinctive spectrum showing a 2:1 ratio of Cu:S suggests the mineral chalcocite (Cu2S). Such a mineral is usually associated with copper sulphide deposits and has not previously been reported for the Sydney coalfields. An extraneous source is suspected, possibly via the stone dust or other mining practice.

20. ibid photo #4. Calcite spectrum CaCO3, with spurious Si and artifact Au.

21. ibid photo #8. Pyrite spectrum.

22. Respirable coal dust on Millipore filter collected at the Brusher station (sample #85-9). Sample has been low temperature ashed on a gold coated glass slide and recoated with gold. Photos 10 and 11 show the extensive flocculation and agglomeration of particles. This spectrum is a composite based on a spot analysis, likely on oxidized pyrite with peak contributions from the glass substrate (Si, Zn) and clays (Al, Si, K). The presence of Mn is enigmatic.

23. ibid. As for the previous spectrum, the particle identity is not discernible because of the substrate interference.

24. Gold coated glass slide: i.e. substrate to photos 10, 11, and EDX #22, 23. Note the abundance of Zn, K, Ti as well as the dominant Si peak. This spectrum is an area scan for 25 seconds.

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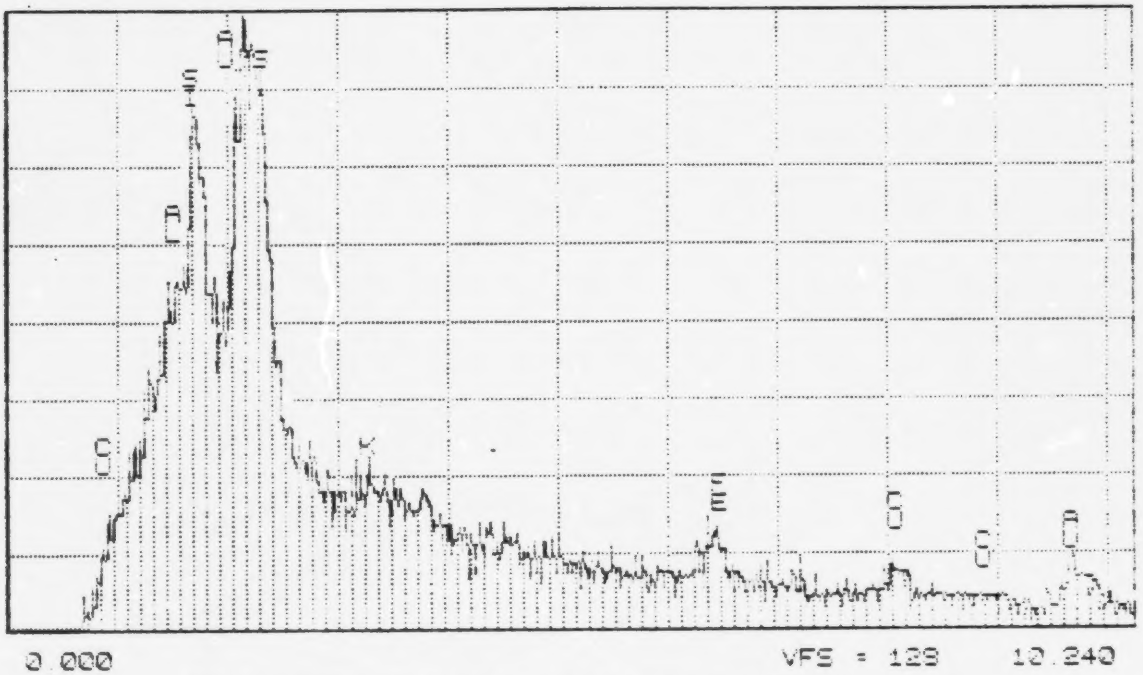

10 DUSTS LTA-COMP1 20KV P0311-01 SPOT #1

1

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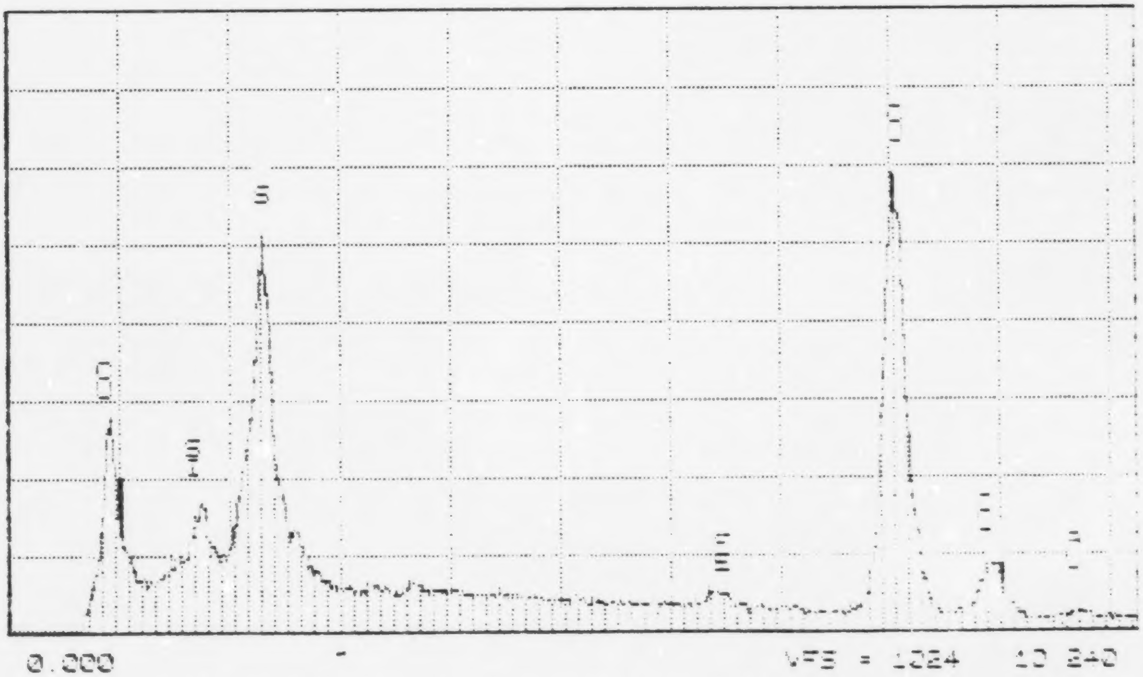

10 DUSTS LTA-COMP1 20KV P0311-01 SPOT #2

2

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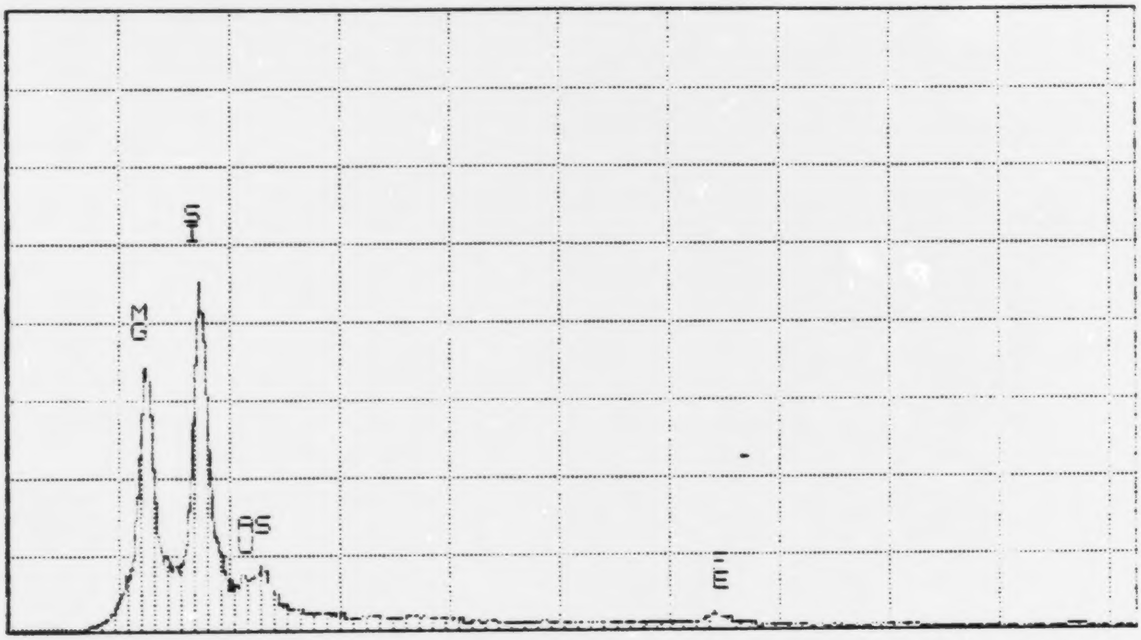

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10 DUSTS LTA-COMP1 20KV P0311-01 SPOT #3

3

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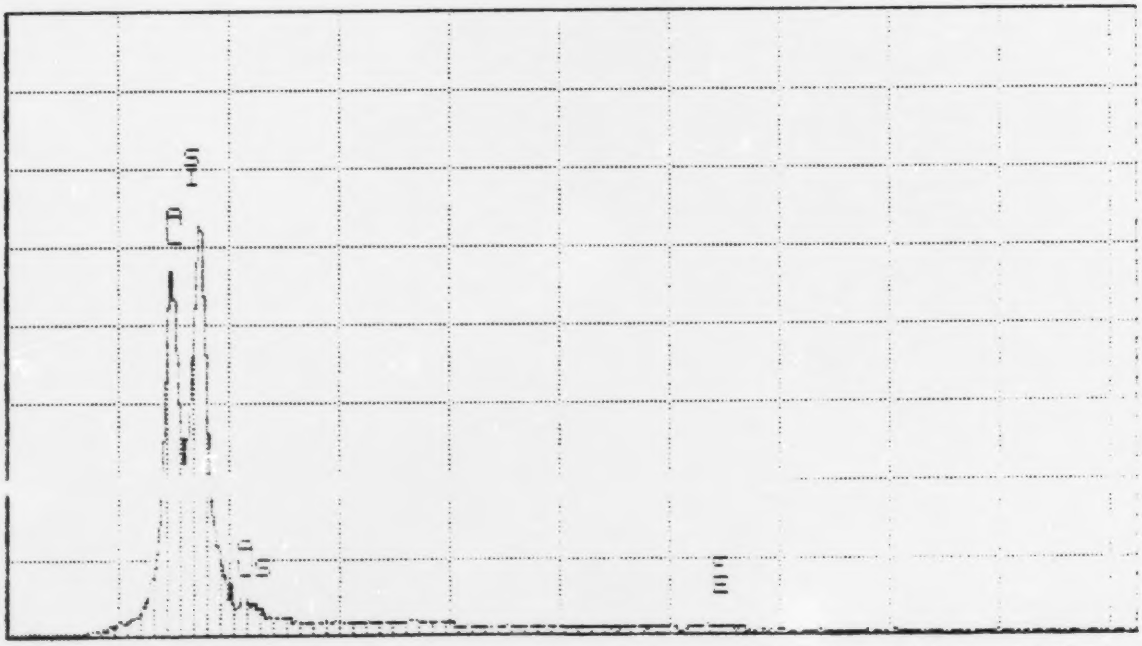

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10 DUSTS LTA-COMP1 20KV P0311-01 SPOT #4

4

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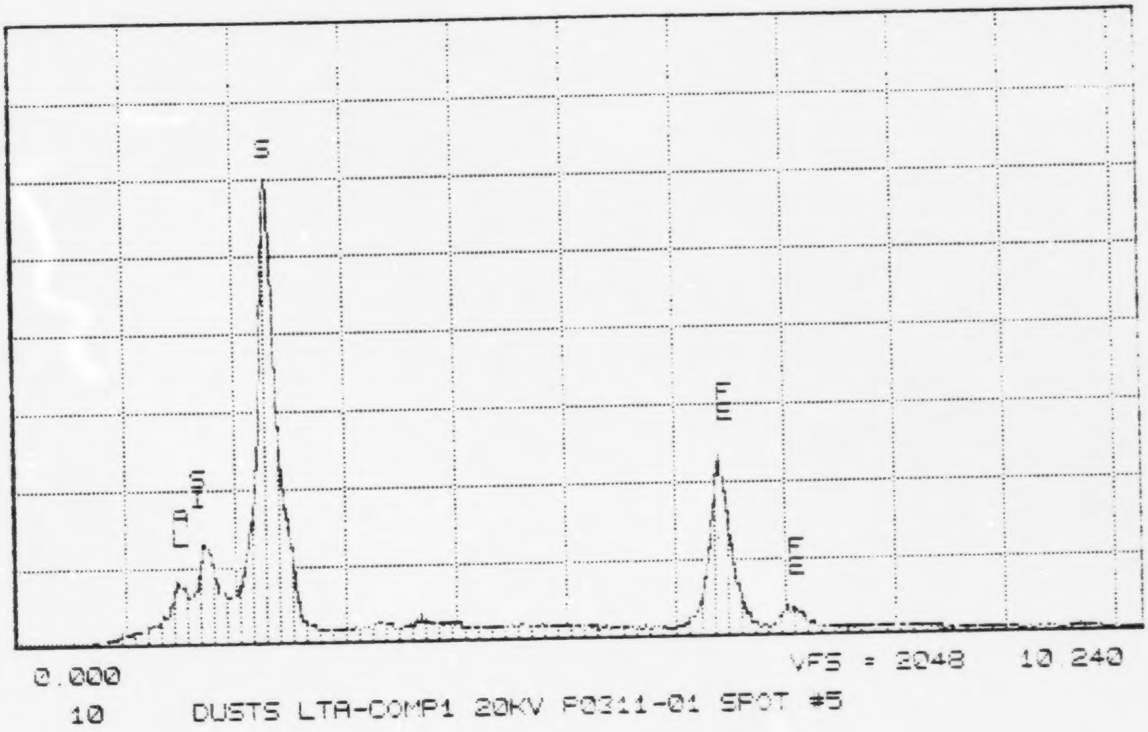

DUSTS LTA-COMP1 20KV P0311-01 SPOT #5

5

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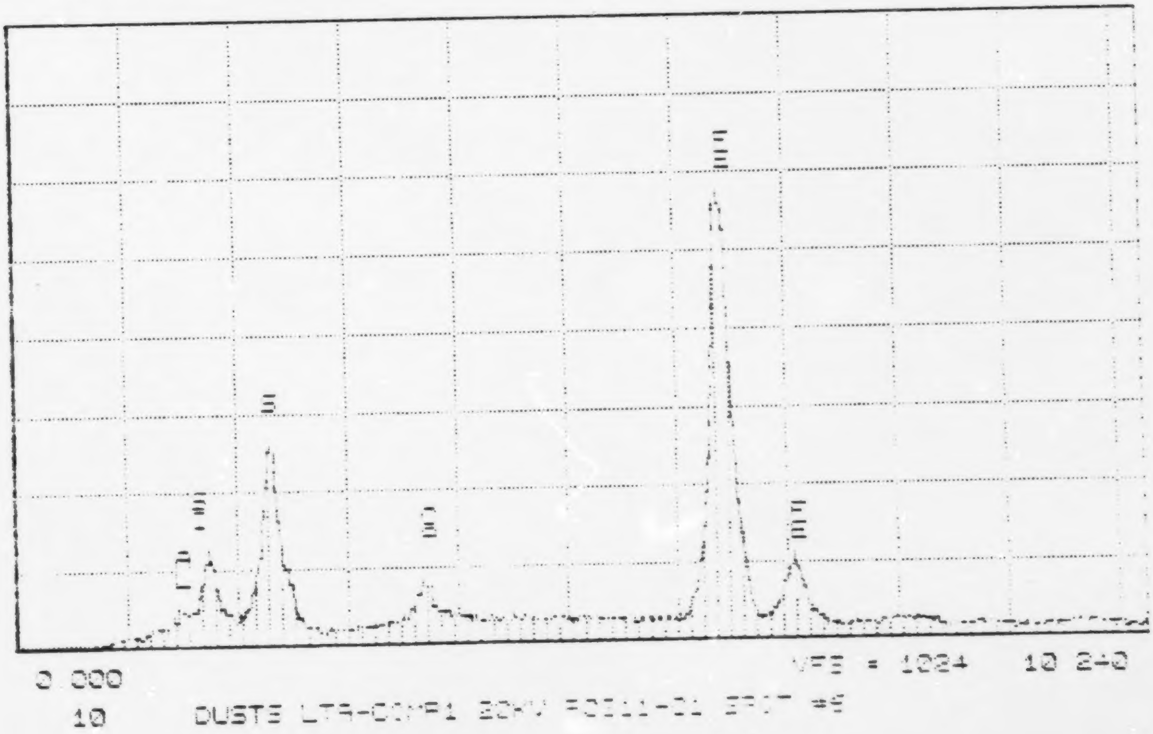

DUSTS LTA-COMP1 20KV P0311-01 SPOT #5

6

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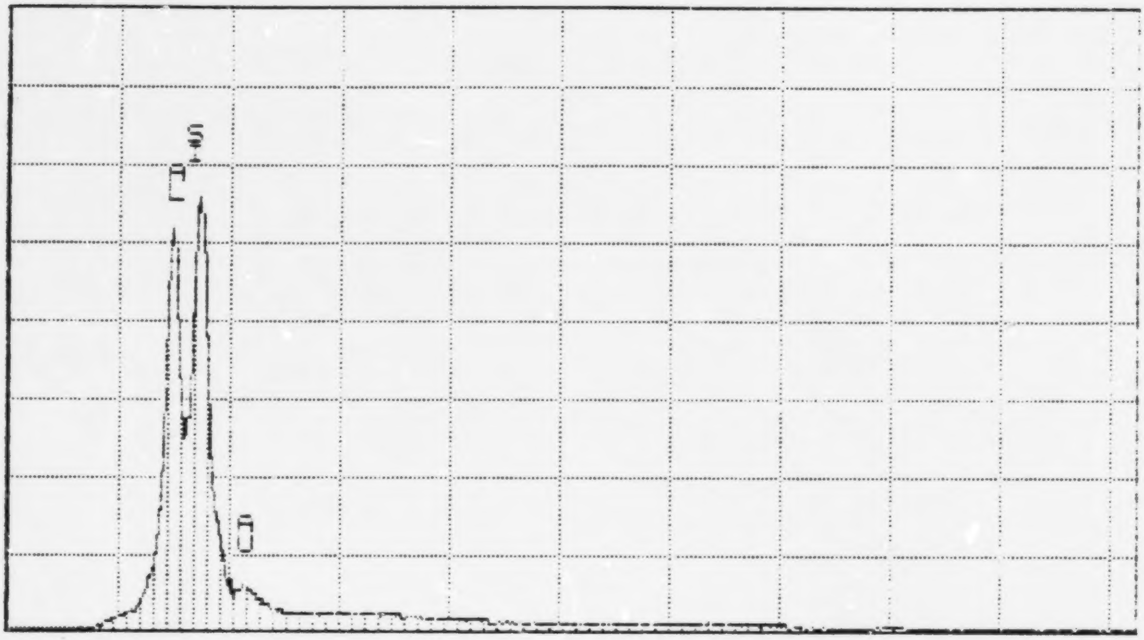

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7

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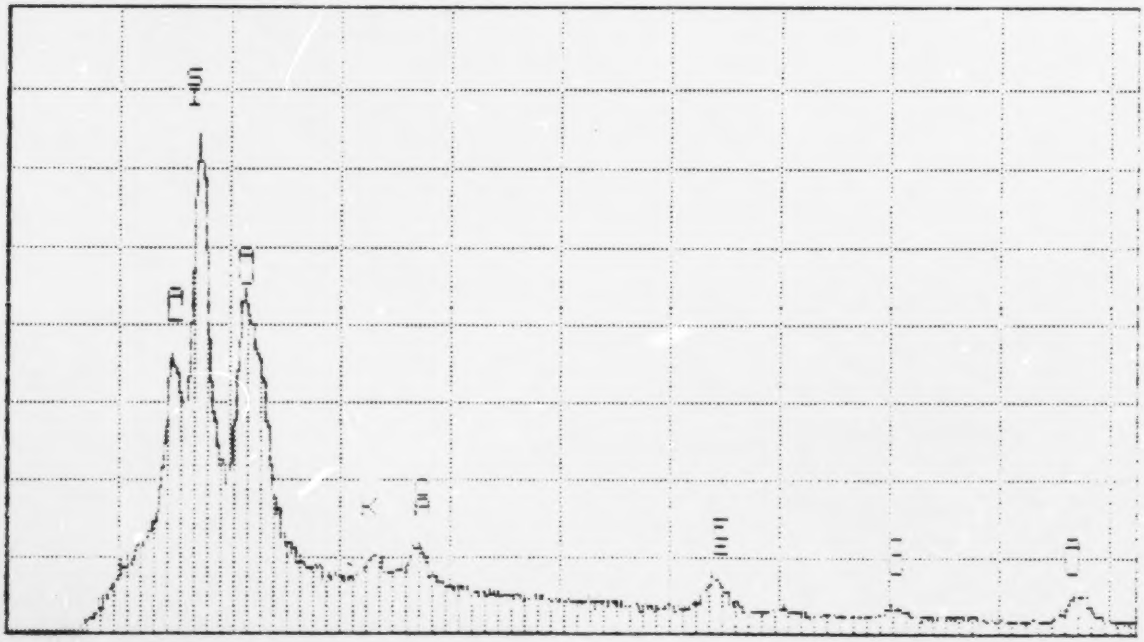

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8

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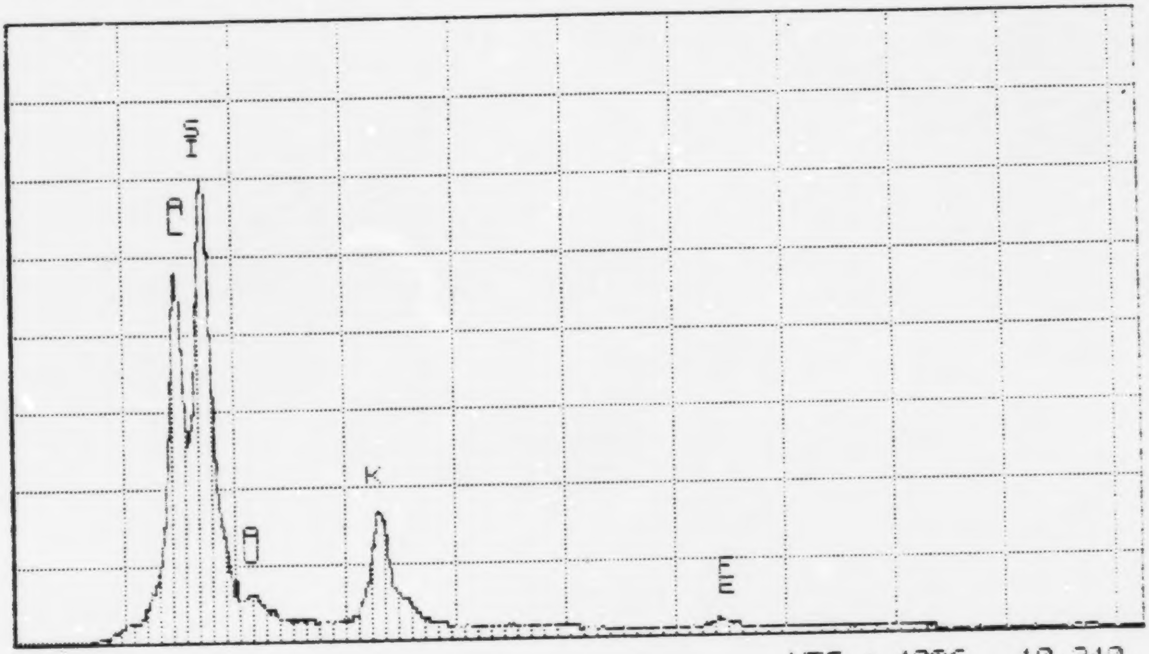

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9

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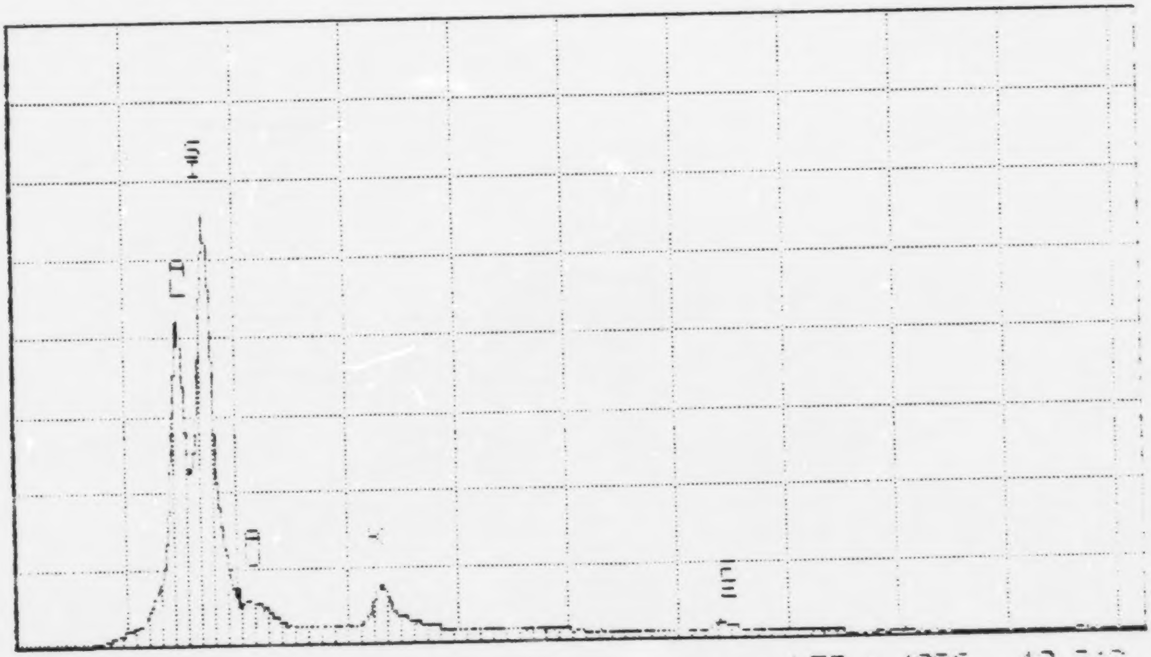

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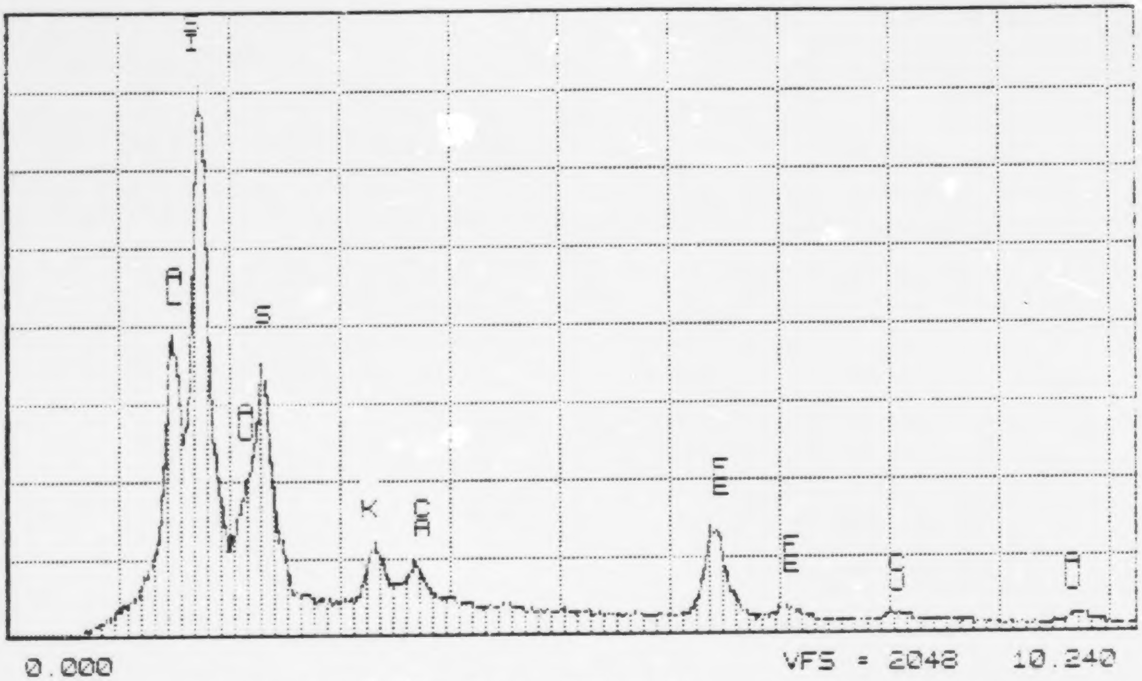

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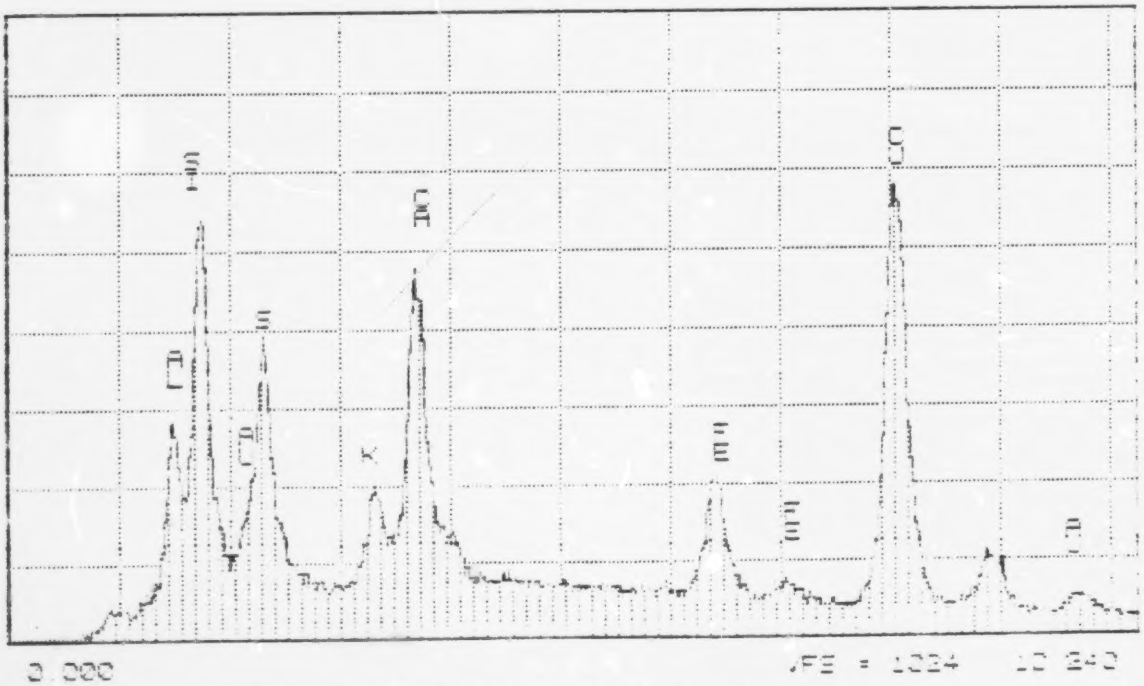

20 DUSTS LTA-COMP1 20KV P0312-01 spot #6

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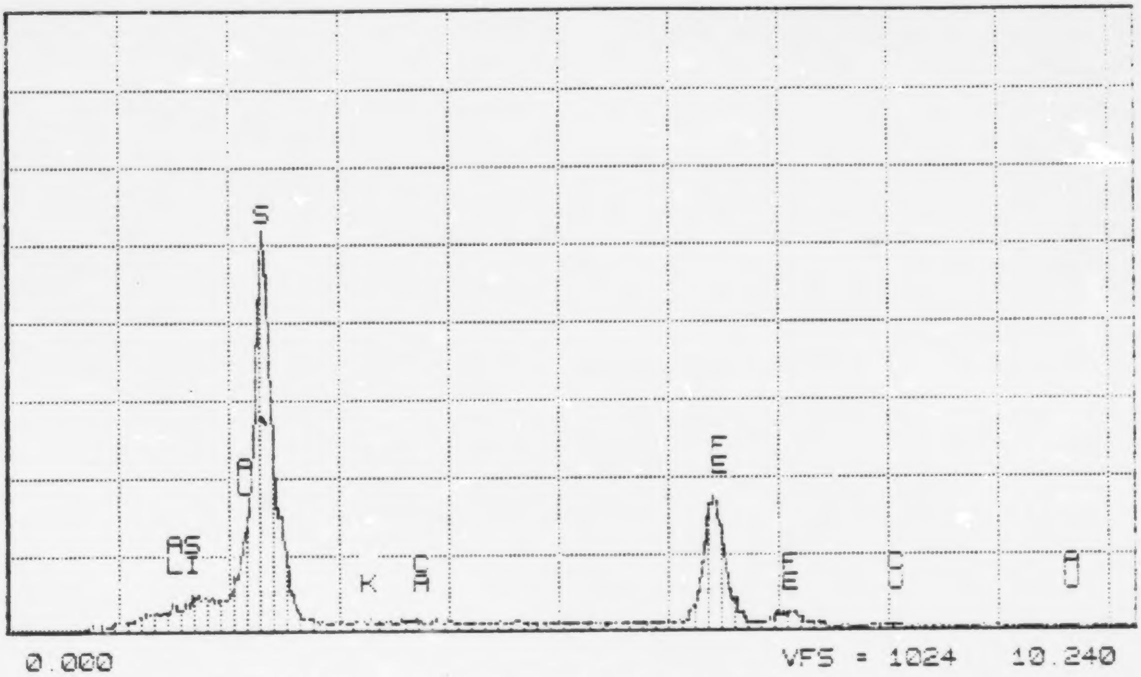

10 DUSTS LTA-COMP1 20KV P0312-02 spot

13

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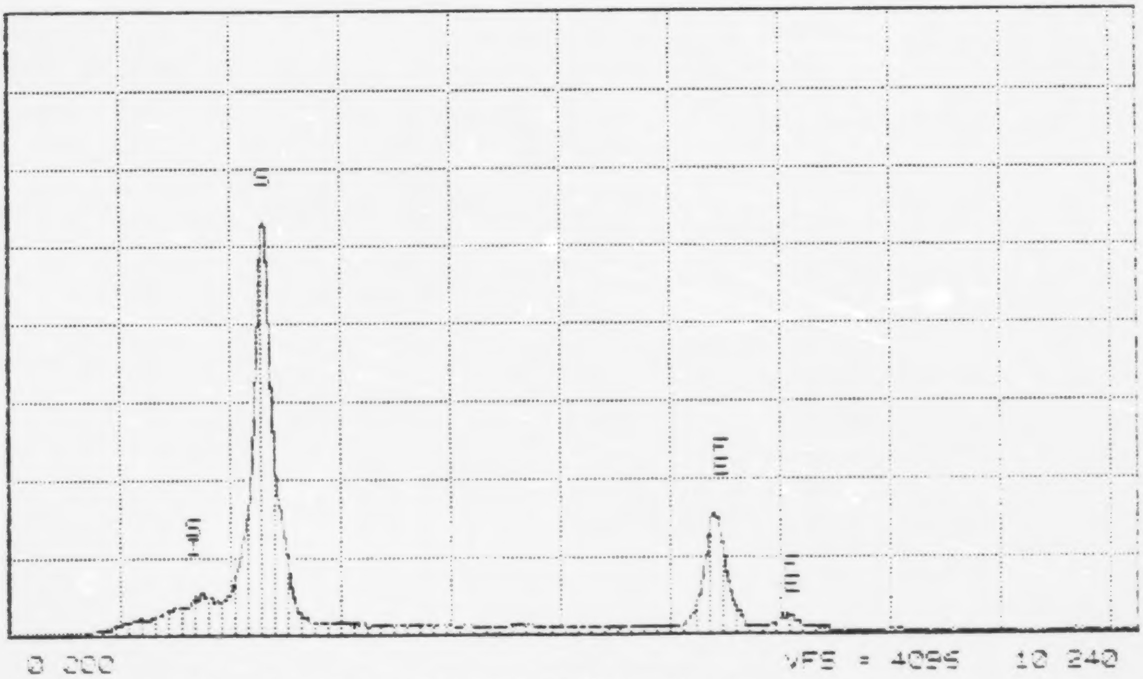

10 DUSTS LTA-COMP1 20KV P0312-02 spot #1

14

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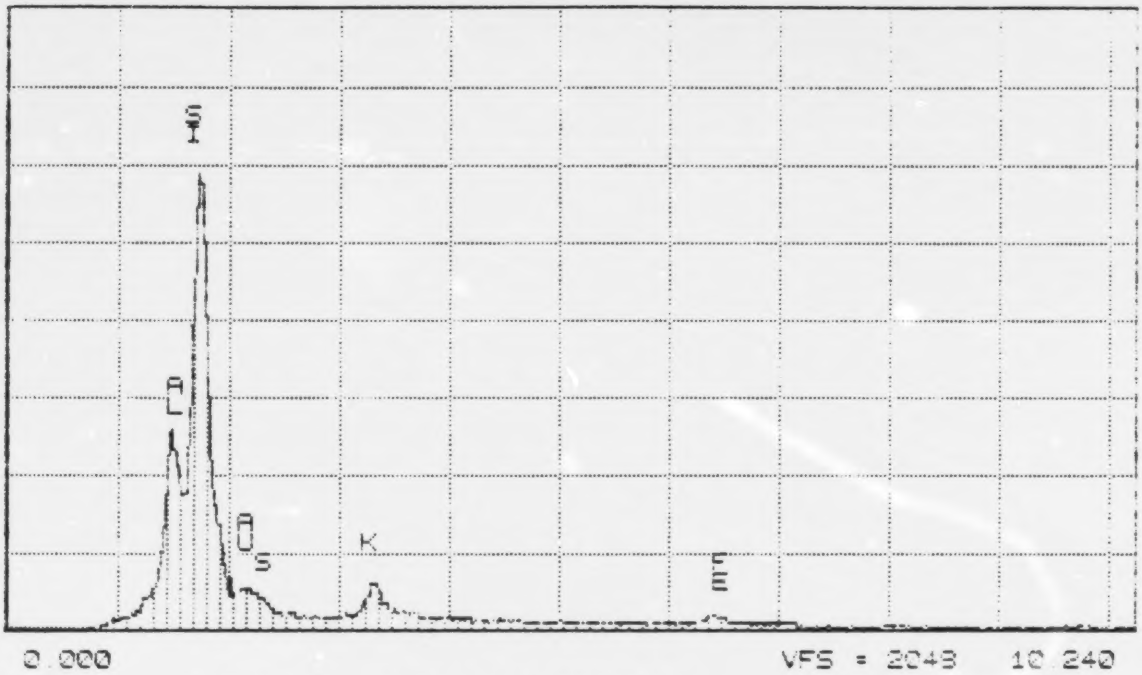

10 DUSTS LTA-COMP1 20KV P0312-03 spct #2

15

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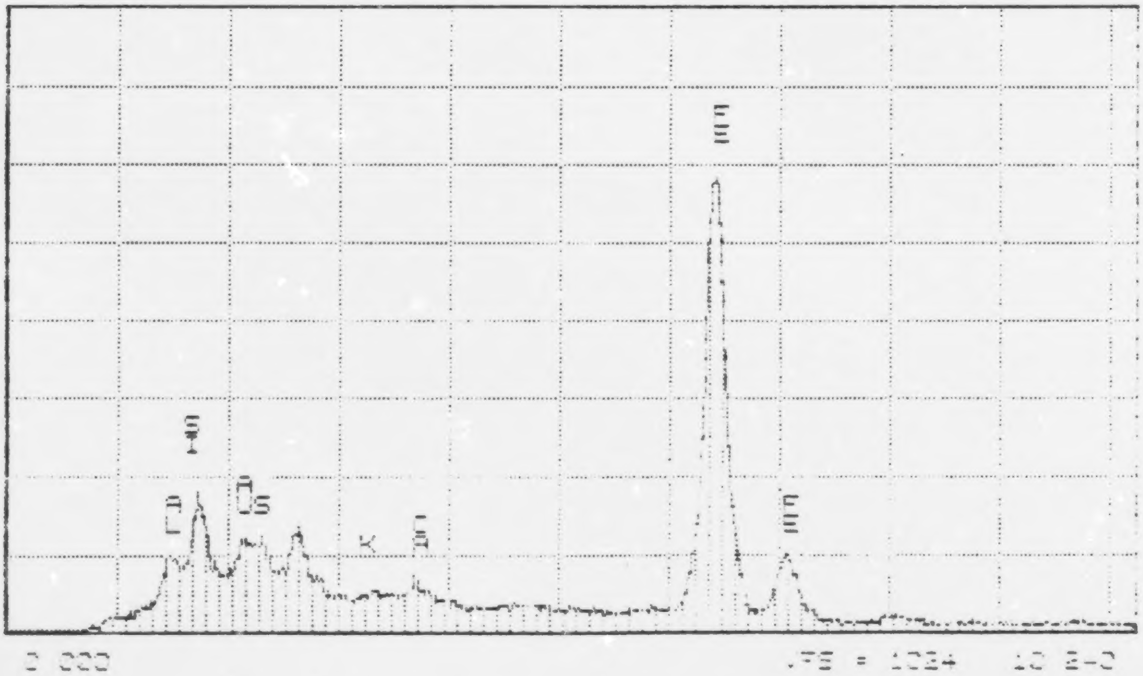

10 DUSTS LTA-COMP1 20KV P0312-03 spct #3

16

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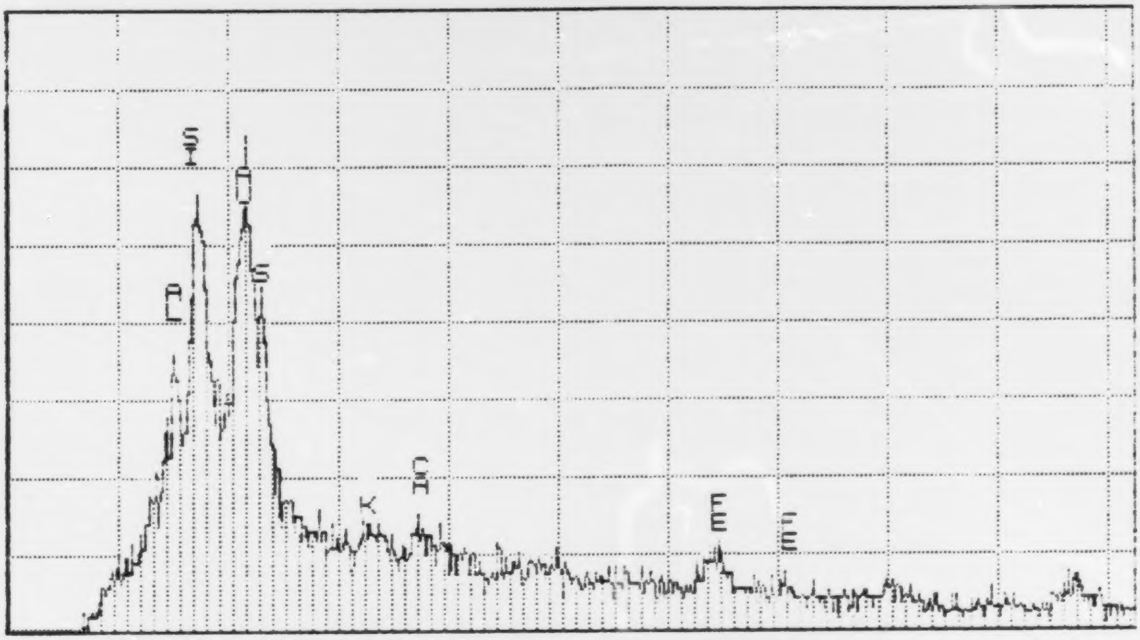

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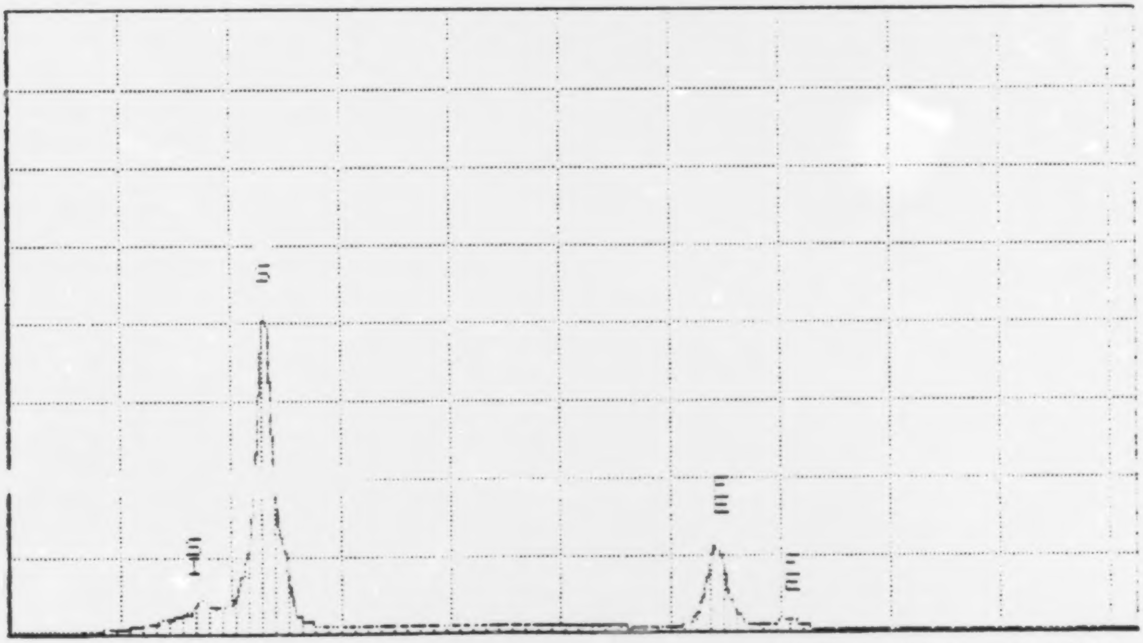

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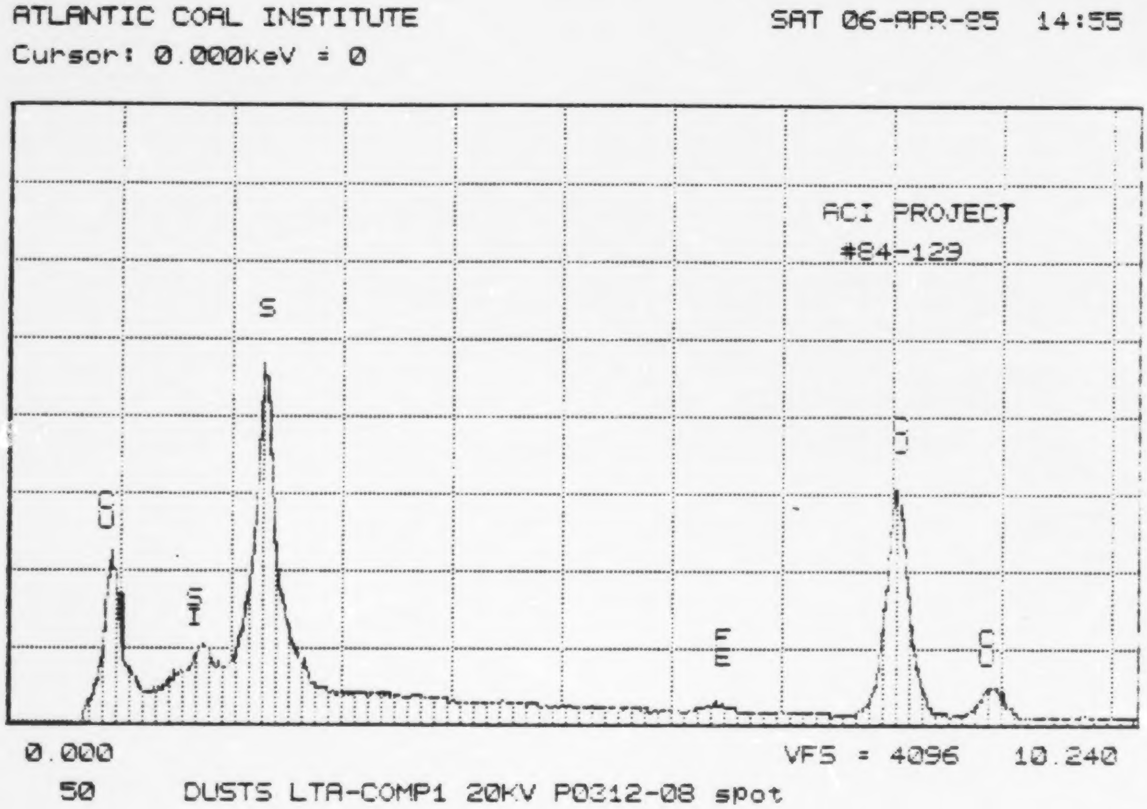
18



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ACI PROJECT
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50 DUSTS LTA-COMP1 20KV P0312-08 spot

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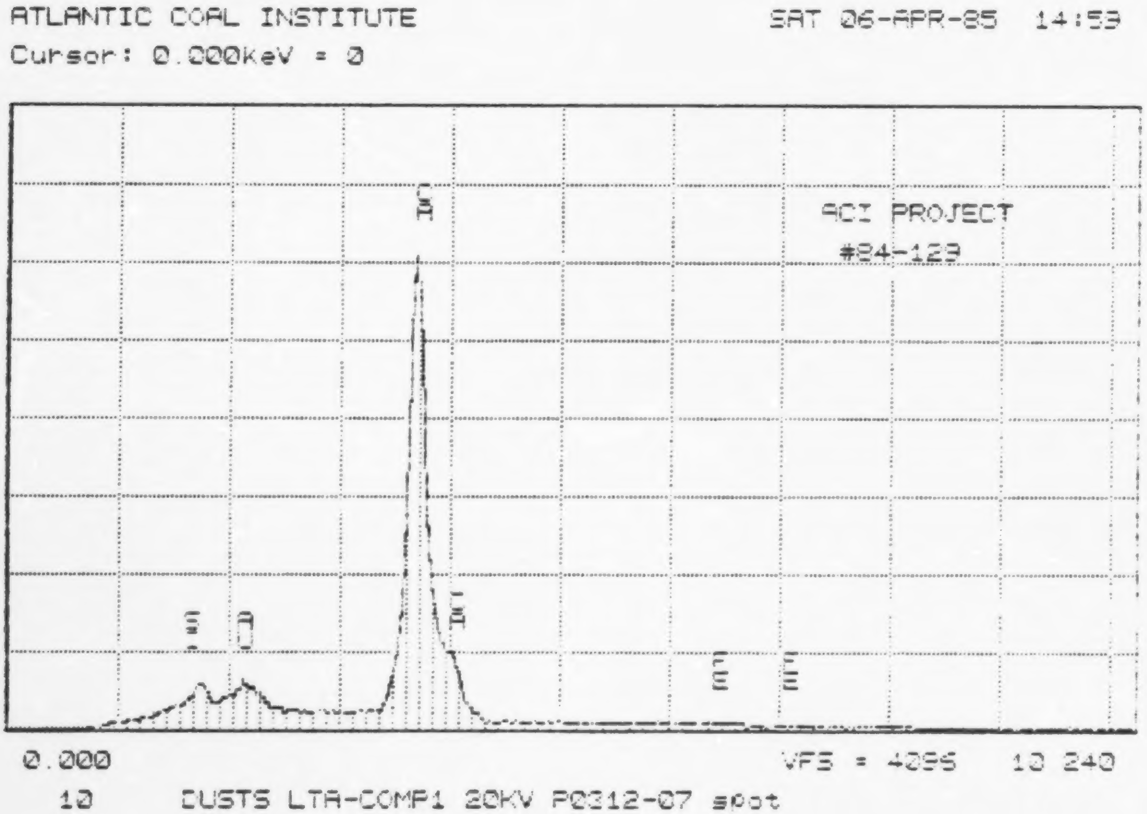

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10 DUSTS LTA-COMP1 20KV P0312-07 spot

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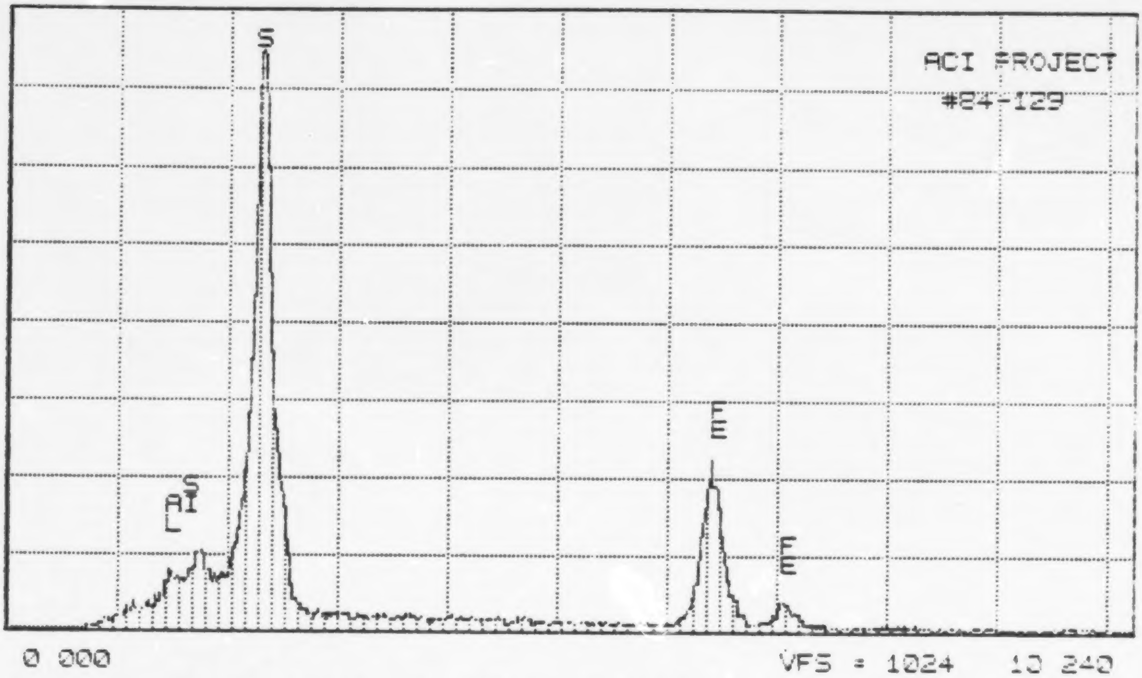

10 DUSTS LTA-COMP1 20KV P0312-09 spot

21

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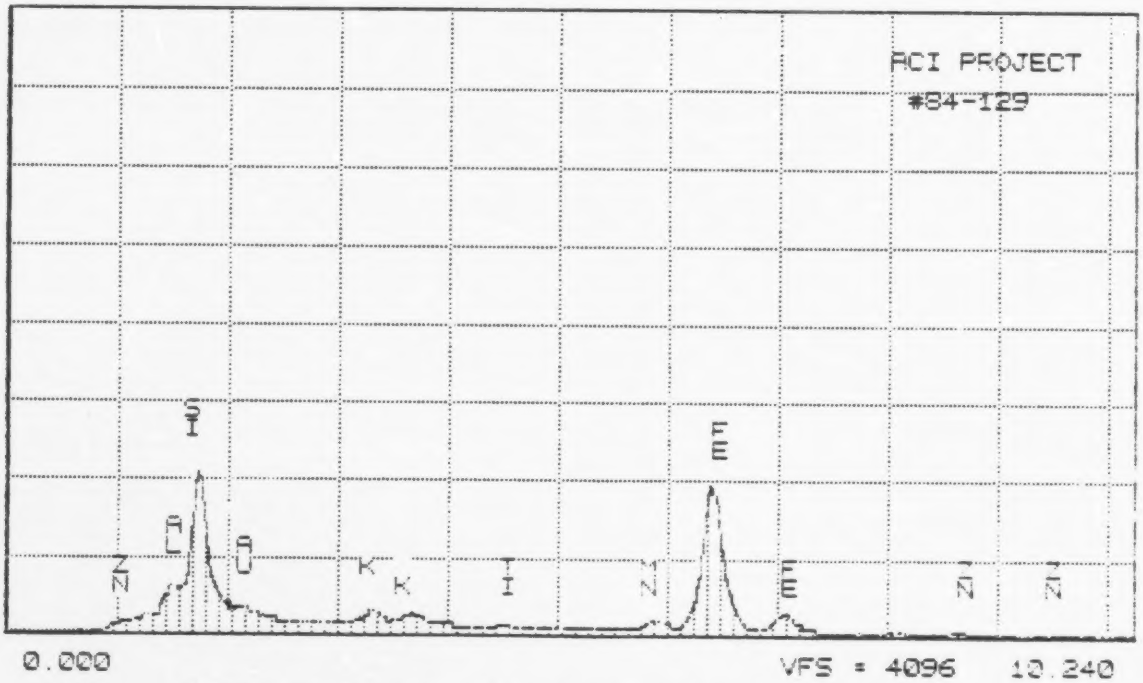

10 DUST LTA FIL AU COAT GLASS SPOT PARTICLE

22

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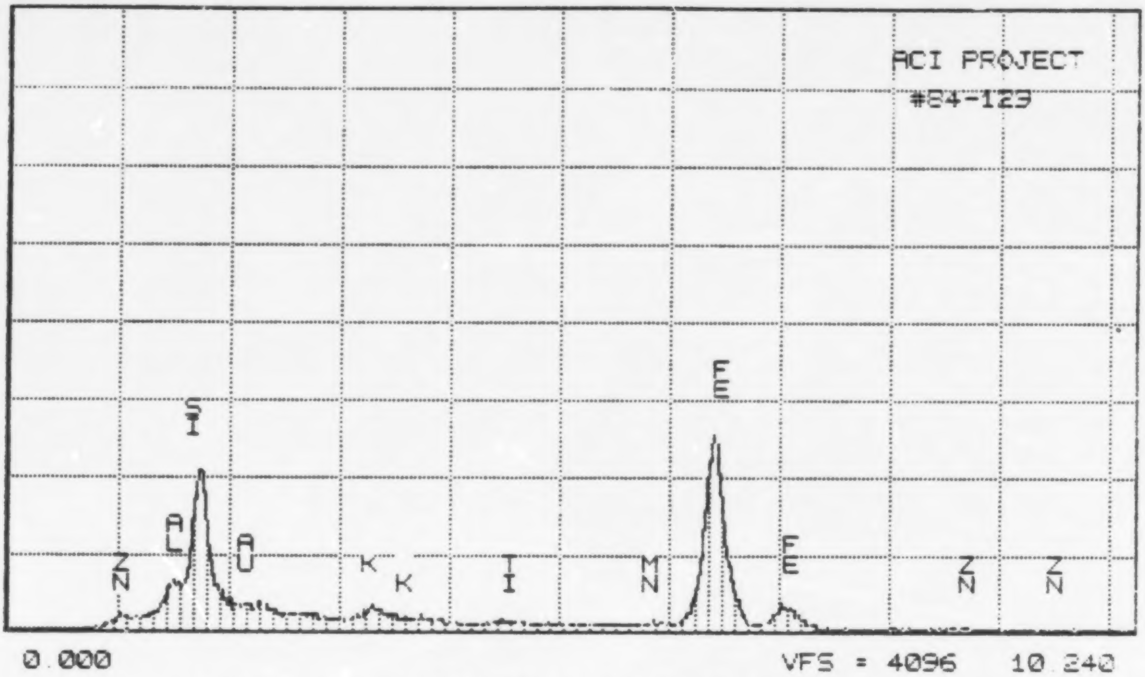

10 DUST LTA FIL AU COAT GLASS SPOT PARTICLE (2)

23

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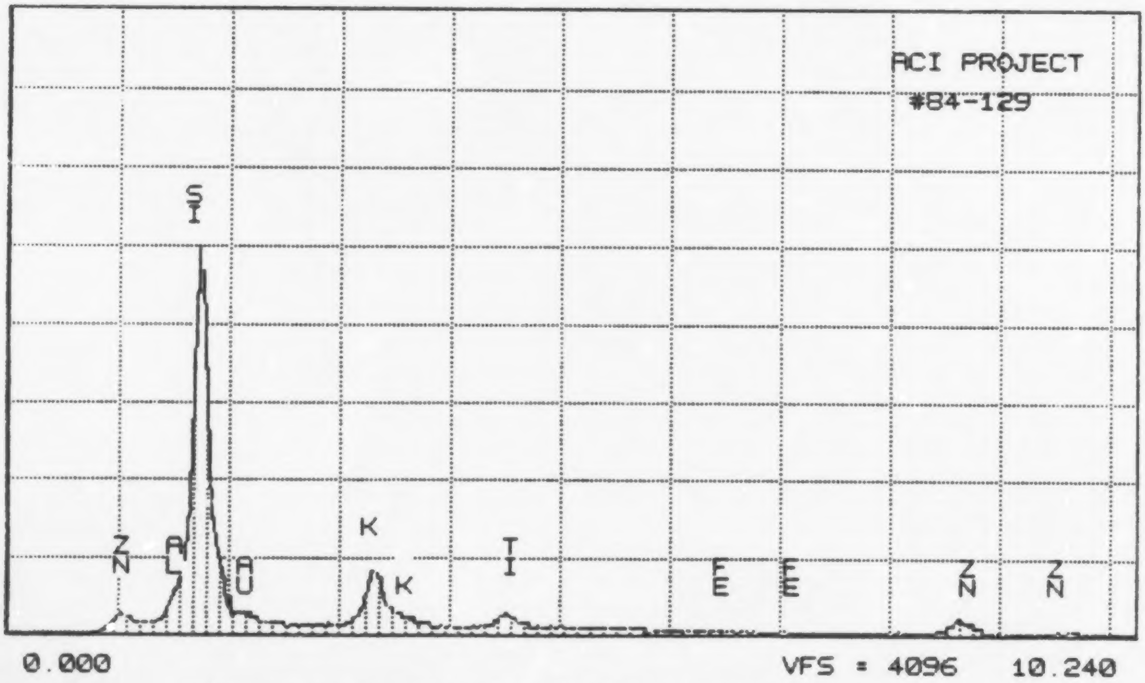

25 DUST LTA FIL AU COAT GLASS R1 BACKGRND 35KX

24

5a. EDX SPECTRA-REPLICATE AREA SCANS

EDX spectra from 100 second area rasters at various magnifications (500x, 1000x, x2000) on the same sample:

Coal dust on Nuclepore filter
(sample #85-15), gold coated.

Data reduction and ZAF correction procedures are based on the Tracor Northern SSQ program for bulk analysis of flat specimens. Conditions for quantitative analysis were not met by this particulate sample, therefore, all results are for qualitative comparison only (see also Table 21 in text).

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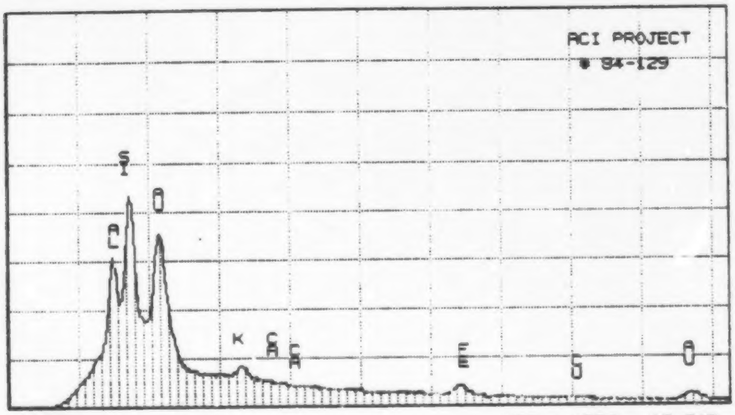

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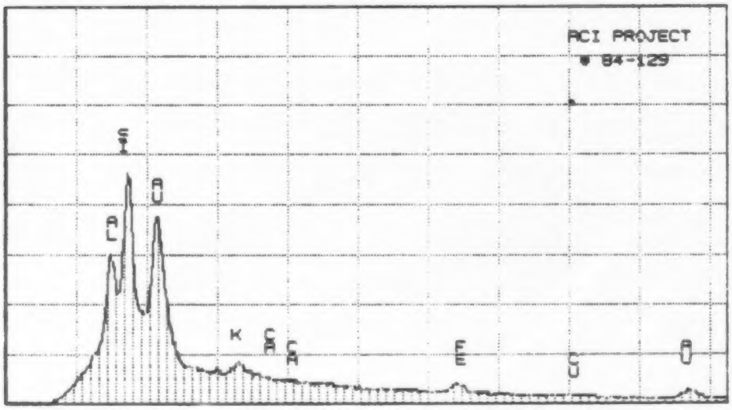

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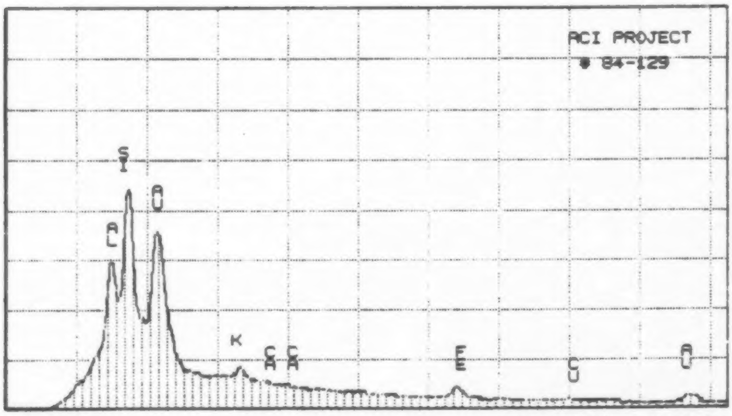

3

0.000 VFS = 4096 10.240
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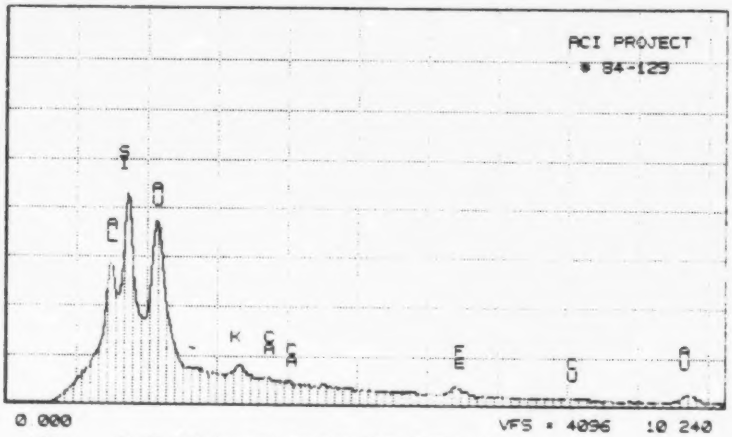

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100 DUST CBCRL #85-15 RAS-EXP 2000x (2)

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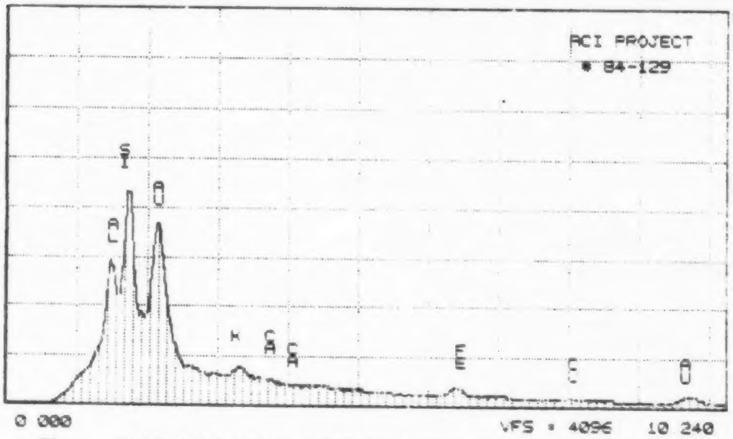

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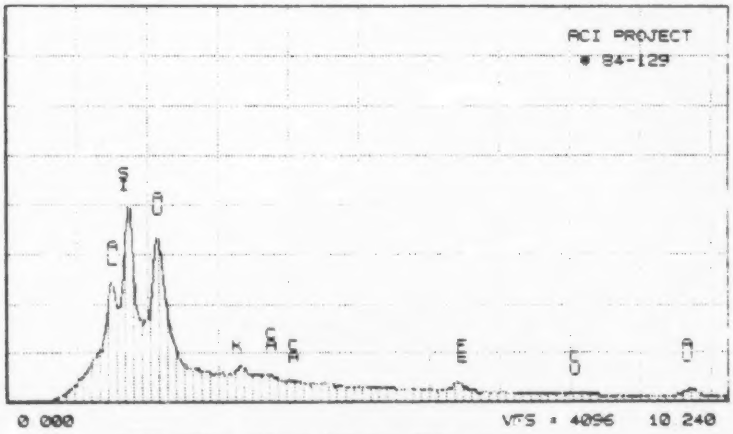

6

100 DUST CBCRL #85-15 RAS-EXP 500x (2)

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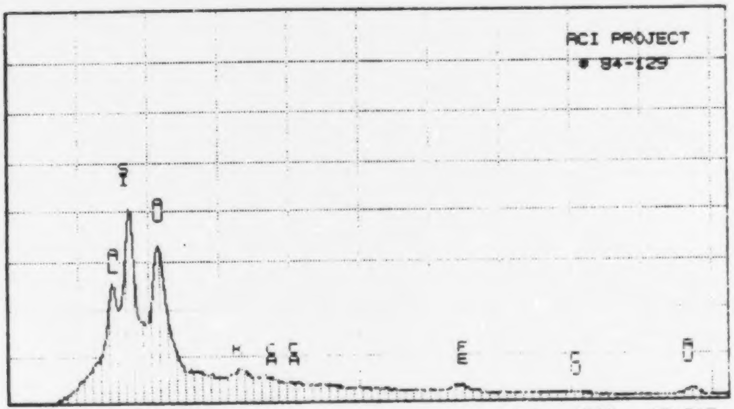

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7

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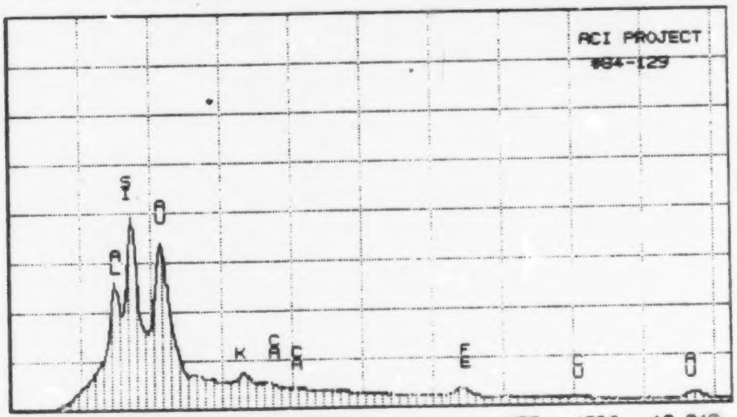

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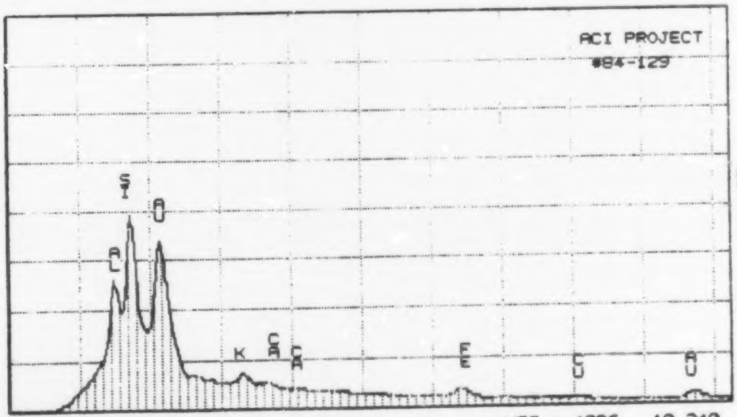

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100 DUST CBCAL #85-15 AAS-EXP 500 X (3)

9

1

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SEMI-QUANTITATIVE ANALYSIS: DUST CBCRL #85-15 RAS-EXP 2000X
EL    NORM. K-RATIO

AL-K 0.24999 +- 0.00232
SI-K 0.40959 +- 0.00284
S -K 0.17673 +- 0.00197
FE-K 0.08322 +- 0.00231
CA-K-0.00000 +- 0.00000
K -K 0.03593 +- 0.00097
CL-K 0.00801 +- 0.00043
TI-K-0.00000 +- 0.00000
CU-K 0.03650 +- 0.00201

ZAF CORRECTION        20.00 KV        30.00 Degs

PT.NO.   1

No. of Iterations  4
----      K       [Z]     [A]     [F]    [ZAF]  ATOM.%  WT.%
AL-K   0.249   0.990   1.336   0.976   1.293   24.00   21.36
SI-K   0.409   0.967   1.672   0.993   1.608   47.20   43.49  *
S -K   0.176   0.991   1.825   0.998   1.806   20.06   21.09  *
FE-K   0.083   1.172   1.031   0.994   1.203    3.60    6.62
CA-K   0.000   1.029   1.278   0.997   1.313    0.00    0.00 G
K -K   0.035   1.050   1.381   0.998   1.448    2.40    3.44
CL-K   0.008   1.030   1.817   0.997   1.869    0.86    0.99  *
TI-K   0.000   1.144   1.135   0.992   1.289    0.00    0.00 G
CU-K   0.036   1.228   1.017   1.000   1.249    1.43    3.01
 * - High Absorbance
```

2

```
SEMI-QUANTITATIVE ANALYSIS: DUST CBCRL #85-15 RAS-EXP 1000X
EL    NORM. K-RATIO

AL-K 0.23798 +- 0.00228
SI-K 0.41514 +- 0.00287
S -K 0.17961 +- 0.00199
FE-K 0.07647 +- 0.00222
CA-K-0.00000 +- 0.00000
K -K 0.03525 +- 0.00097
CL-K 0.00666 +- 0.00039
TI-K-0.00000 +- 0.00000
CU-K 0.04885 +- 0.00234

ZAF CORRECTION        20.00 KV        30.00 Degs

PT.NO.   2

No. of Iterations  1
----      K       [Z]     [A]     [F]    [ZAF]  ATOM.%  WT.%
AL-K   0.237   0.991   1.346   0.975   1.301   23.14   20.45
SI-K   0.415   0.966   1.662   0.993   1.596   47.80   43.81  *
S -K   0.179   0.990   1.827   0.998   1.806   20.46   21.43  *
FE-K   0.076   1.173   1.031   0.992   1.201    3.31    6.07
CA-K   0.000   1.033   1.290   0.997   1.330    0.00    0.00 G
K -K   0.035   1.050   1.385   0.998   1.452    2.65    3.38
CL-K   0.006   1.031   1.829   0.998   1.882    0.72    0.83  *
TI-K   0.000   1.149   1.141   0.993   1.303    0.00    0.00 G
CU-K   0.048   1.229   1.015   1.000   1.248    1.92    4.03
 * - High Absorbance
```

3

```
SEMI-QUANTITATIVE ANALYSIS: DUST CBCRL #85-15 RAS-EXP 500X
EL    NORM. K-RATIO

AL-K 0.23666 +- 0.00221
SI-K 0.41083 +- 0.00278
S -K 0.17964 +- 0.00194
FE-K 0.08360 +- 0.00226
CA-K-0.00000 +- 0.00000
K -K 0.03365 +- 0.00092
CL-K 0.01019 +- 0.00047
TI-K-0.00000 +- 0.00000
CU-K 0.04538 +- 0.00219

ZAF CORRECTION        20.00 KV        30.00 Degs

PT.NO.   3

No. of Iterations  1
----      K       [Z]     [A]     [F]    [ZAF]  ATOM.%  WT.%
AL-K   0.236   0.990   1.350   0.975   1.304   23.13   20.41
SI-K   0.410   0.966   1.662   0.993   1.596   47.38   43.35  *
S -K   0.179   0.989   1.819   0.998   1.798   20.42   21.36  *
FE-K   0.083   1.172   1.031   0.993   1.201    3.63    6.64
CA-K   0.000   1.033   1.289   0.997   1.329    0.00    0.00 G
K -K   0.033   1.049   1.386   0.998   1.453    2.54    3.23
CL-K   0.010   1.030   1.823   0.998   1.876    1.11    1.26  *
TI-K   0.000   1.148   1.141   0.993   1.302    0.00    0.00 G
CU-K   0.045   1.228   1.016   1.000   1.248    1.79    3.75
 * - High Absorbance
```

```
SEMI-QUANTITATIVE ANALYSIS: DUST CBCRL #85-15 RAS-EXP 2000X (2)          4
EL     NORM. K-RATIO

AL-K 0.23557 +- 0.00226
SI-K 0.39833 +- 0.00280
S -K 0.18901 +- 0.00203
FE-K 0.08031 +- 0.00227
CA-K 0.00009 +- 0.00005
K -K 0.03524 +- 0.00097
CL-K 0.01411 +- 0.00057
TI-K-0.00000 +- 0.00000
CU-K 0.04729 +- 0.00229

ZAF CORRECTION       20.00 KV       30.00 Degs

PT.NO.   4

No. of Iterations  1
----     K       [Z]     [A]     [F]    [ZAF]  ATOM.%  WT.%
AL-K  0.235  0.990  1.352  0.975  1.307  23.12  20.35
SI-K  0.398  0.965  1.662  0.993  1.594  46.01  42.00  *
S -K  0.189  0.989  1.800  0.997  1.778  21.30  22.22  *
FE-K  0.080  1.172  1.032  0.992  1.201   3.49   6.38
CA-K  0.000  1.032  1.293  0.997  1.332   0.01   0.01
K -K  0.035  1.049  1.389  0.998  1.456   2.67   3.39
CL-K  0.014  1.030  1.823  0.998  1.874   1.53   1.75  *
TI-K  0.000  1.147  1.143  0.993  1.304   0.00   0.00 G
CU-K  0.047  1.227  1.016  1.000  1.248   1.87   3.90
 * - High Absorbance
```

```
SEMI-QUANTITATIVE ANALYSIS: DUST CBCRL #85-15 RAS-EXP 1000X (2)          5
EL     NORM. K-RATIO

AL-K 0.23808 +- 0.00223
SI-K 0.40315 +- 0.00277
S -K 0.18681 +- 0.00199
FE-K 0.08145 +- 0.00224
CA-K-0.00000 +- 0.00000
K -K 0.02945 +- 0.00087
CL-K 0.01683 +- 0.00061
TI-K-0.00000 +- 0.00000
CU-K 0.04419 +- 0.00218

ZAF CORRECTION       20.00 KV       30.00 Degs

PT.NO.   5

No. of Iterations  1
----     K       [Z]     [A]     [F]    [ZAF]  ATOM.%  WT.%
AL-K  0.238  0.990  1.344  0.975  1.299  23.16  20.43
SI-K  0.403  0.966  1.664  0.993  1.597  46.46  42.51  *
S -K  0.186  0.990  1.806  0.997  1.785  21.05  22.02  *
FE-K  0.081  1.172  1.032  0.993  1.202   3.53   6.47
CA-K  0.000  1.033  1.291  0.997  1.331   0.00   0.00 G
K -K  0.029  1.049  1.392  0.998  1.459   2.23   2.84
CL-K  0.016  1.030  1.822  0.998  1.875   1.82   2.09  *
TI-K  0.000  1.148  1.141  0.993  1.303   0.00   0.00 G
CU-K  0.044  1.228  1.016  1.000  1.248   1.74   3.65
 * - High Absorbance
```

```
SEMI-QUANTITATIVE ANALYSIS: DUST CBCRL #85-15 RAS-EXP 500X (2)          6
EL     NORM. K-RATIO

AL-K 0.23835 +- 0.00225
SI-K 0.40988 +- 0.00281
S -K 0.18258 +- 0.00198
FE-K 0.08864 +- 0.00236
CA-K-0.00000 +- 0.00000
K -K 0.03274 +- 0.00092
CL-K 0.01191 +- 0.00052
TI-K-0.00000 +- 0.00000
CU-K 0.03586 +- 0.00198

ZAF CORRECTION       20.00 KV       30.00 Degs

PT.NO.   6

No. of Iterations  2
----     K       [Z]     [A]     [F]    [ZAF]  ATOM.%  WT.%
AL-K  0.238  0.991  1.336  0.975  1.291  23.09  20.41
SI-K  0.409  0.967  1.654  0.993  1.589  47.14  43.20  *
S -K  0.182  0.990  1.813  0.998  1.793  20.72  21.70  *
FE-K  0.088  1.173  1.031  0.994  1.204   3.86   7.08
CA-K  0.000  1.033  1.290  0.997  1.330   0.00   0.00 G
K -K  0.032  1.050  1.387  0.998  1.455   2.47   3.16
CL-K  0.011  1.031  1.823  0.998  1.876   1.29   1.48  *
TI-K  0.000  1.149  1.141  0.993  1.303   0.00   0.00 G
CU-K  0.035  1.229  1.017  1.000  1.250   1.42   2.97
 * - High Absorbance
```

7

```
SEMI-QUANTITATIVE ANALYSIS: DUST CBCRL #85-15 RAS-EXP 2000X (3)
EL    NORM. K-RATIO

AL-K 0.22680 +- 0.00232
SI-K 0.41805 +- 0.00301
S -K 0.18329 +- 0.00210
FE-K 0.08231 +- 0.00241
CA-K 0.00503 +- 0.00039
K -K 0.03045 +- 0.00094
CL-K 0.00921 +- 0.00048
TI-K-0.00000 +- 0.00000
CU-K 0.04483 +- 0.00234

ZAF CORRECTION        20.00 KV        30.00 Degs

PT.NO.   7

No. of Iterations  1
----     K       [Z]     [A]     [F]    [ZAF]   ATOM.%  WT.%
AL-K  0.226  0.990  1.349  0.974  1.303  22.23  19.60
SI-K  0.418  0.966  1.644  0.993  1.578  47.87  43.77  *
S -K  0.183  0.990  1.817  0.998  1.796  20.89  21.83  *
FE-K  0.082  1.173  1.032  0.993  1.202   3.59   6.56
CA-K  0.005  1.033  1.288  0.997  1.328   0.34   0.44
K -K  0.030  1.050  1.386  0.998  1.453   2.30   2.93
CL-K  0.009  1.030  1.827  0.998  1.880   1.00   1.15  *
TI-K  0.000  1.148  1.142  0.993  1.304   0.00   0.00 G
CU-K  0.044  1.228  1.016  1.000  1.249   1.78   3.71
 * - High Absorbance
```

8

```
SEMI-QUANTITATIVE ANALYSIS: DUST CBCRL #85-15 RAS-EXP 1000X (3)
EL    NORM. K-RATIO

AL-K 0.23024 +- 0.00234
SI-K 0.40527 +- 0.00297
S -K 0.18926 +- 0.00214
FE-K 0.09108 +- 0.00254
CA-K 0.00046 +- 0.00012
K -K 0.02698 +- 0.00089
CL-K 0.01682 +- 0.00066
TI-K-0.00000 +- 0.00000
CU-K 0.03985 +- 0.00221

ZAF CORRECTION        20.00 KV        30.00 Degs

PT.NO.   8

No. of Iterations  1
----     K       [Z]     [A]     [F]    [ZAF]   ATOM.%  WT.%
AL-K  0.230  0.990  1.350  0.975  1.304  22.60  19.89
SI-K  0.405  0.965  1.652  0.993  1.584  46.56  42.51  *
S -K  0.189  0.989  1.801  0.998  1.779  21.30  22.31  *
FE-K  0.091  1.172  1.032  0.993  1.202   3.97   7.25
CA-K  0.000  1.032  1.290  0.997  1.329   0.03   0.04
K -K  0.026  1.049  1.393  0.998  1.459   2.05   2.61
CL-K  0.016  1.030  1.825  0.998  1.877   1.83   2.09  *
TI-K  0.000  1.148  1.141  0.993  1.301   0.00   0.00 G
CU-K  0.039  1.227  1.017  1.000  1.249   1.58   3.30
 * - High Absorbance
```

9

```
SEMI-QUANTITATIVE ANALYSIS: DUST CBCRL #85-15 RAS-EXP 500X (3)
EL    NORM. K-RATIO

AL-K 0.23024 +- 0.00234
SI-K 0.40527 +- 0.00297
S -K 0.18926 +- 0.00214
FE-K 0.09108 +- 0.00254
CA-K 0.00046 +- 0.00012
K -K 0.02698 +- 0.00089
CL-K 0.01682 +- 0.00066
TI-K-0.00000 +- 0.00000
CU-K 0.03985 +- 0.00221

ZAF CORRECTION        20.00 KV        30.00 Degs

PT.NO.  10

No. of Iterations  0
----     K       [Z]     [A]     [F]    [ZAF]   ATOM.%  WT.%
AL-K  0.230  0.990  1.350  0.975  1.304  22.59  19.89
SI-K  0.405  0.965  1.651  0.993  1.584  46.57  42.52  *
S -K  0.189  0.989  1.802  0.998  1.779  21.30  22.31  *
FE-K  0.091  1.172  1.031  0.993  1.202   3.97   7.25
CA-K  0.000  1.032  1.289  0.997  1.329   0.03   0.04
K -K  0.026  1.049  1.392  0.998  1.459   2.05   2.61
CL-K  0.016  1.030  1.824  0.998  1.876   1.83   2.09  *
TI-K  0.000  1.148  1.141  0.993  1.301   0.00   0.00 G
CU-K  0.039  1.228  1.017  1.000  1.249   1.58   3.30
 * - High Absorbance
```

5b. EDX SPECTRA - AREA SCANS ON COAL DUST-LOADED FILTERS

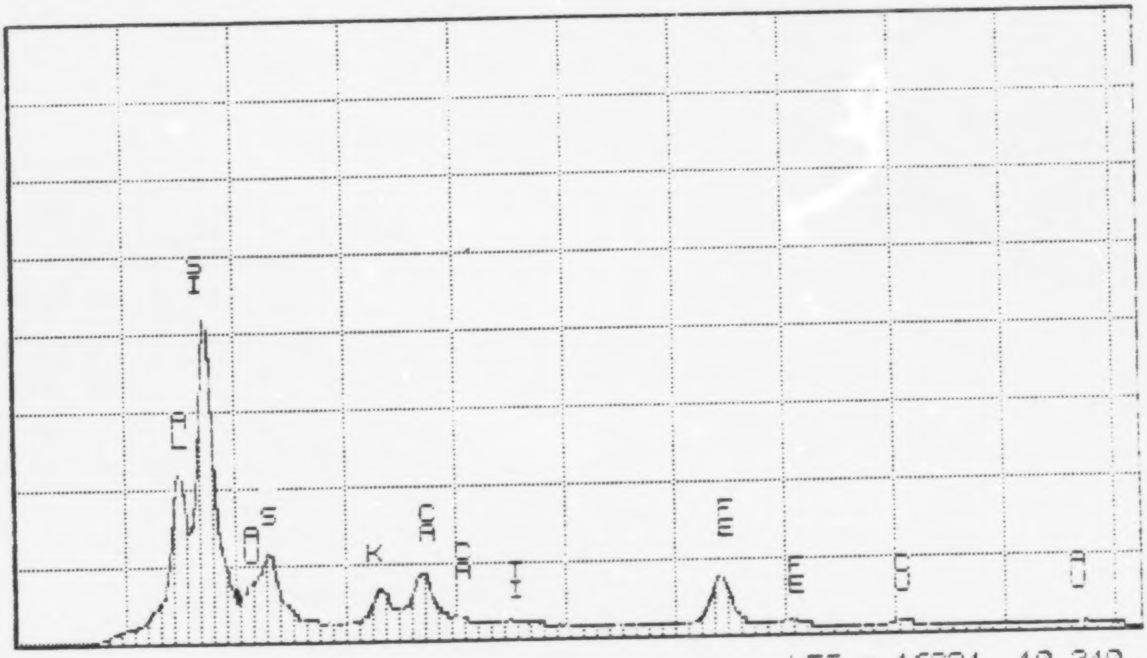
EDX spectra from 200 second area rasters at various magnifications on coal dust-loaded filters. A summary of this data is given in Table 22 (text).

Notes:

1. LTA comp: The removal of coal macerals by oxygen plasma ashing results in a much reduced xray continuum background (EDX #1).
2. Filter #85-15: Gold coating in EDX #11 masks the presence of S as shown by a carbon coated portion of this filter (EDX #10).
3. Data reduction and ZAF correction procedures are from Tracor Northern SSQ programme and do not conform with these particulate samples.
4. EDX #9 shows hatched regions selected as background for all SSQ data reduction.

ATLANTIC COAL INSTITUTE WED 03-APR-85 22:37

Cursor: 0.000keV = 0



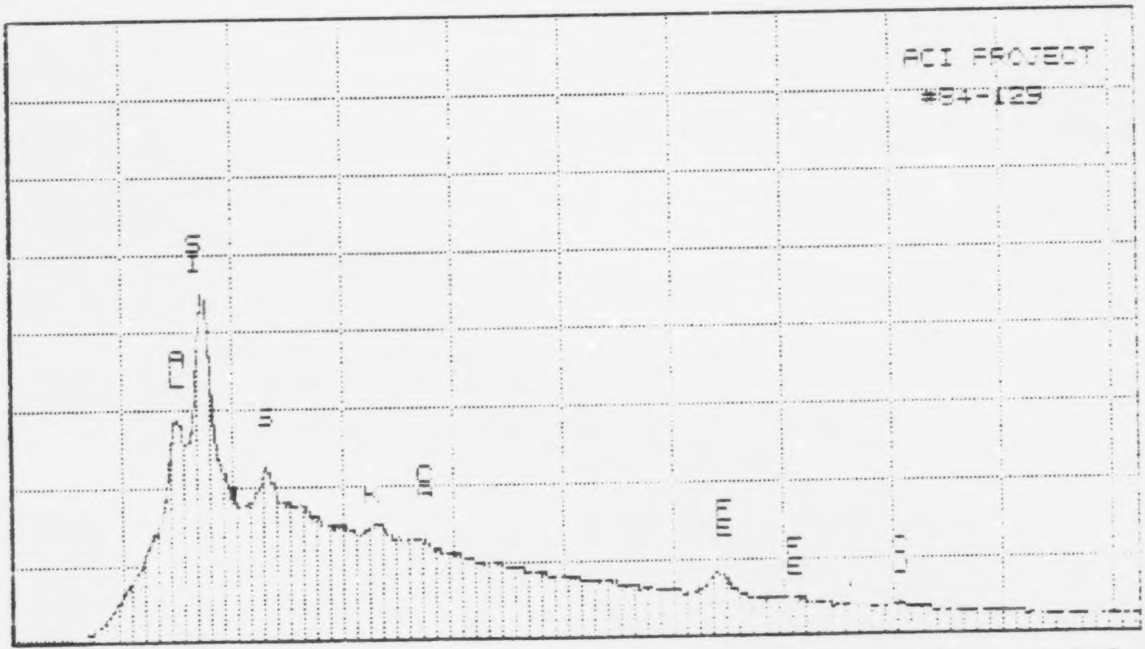
1

0.000 VFS = 16384 10.240

200 DUSTS LTA-COMP1 20KV AREA 500X (SUM 4 PTS)

ATLANTIC COAL INSTITUTE WED 27-MAR-85 08:54

Cursor: 0.010keV = 0

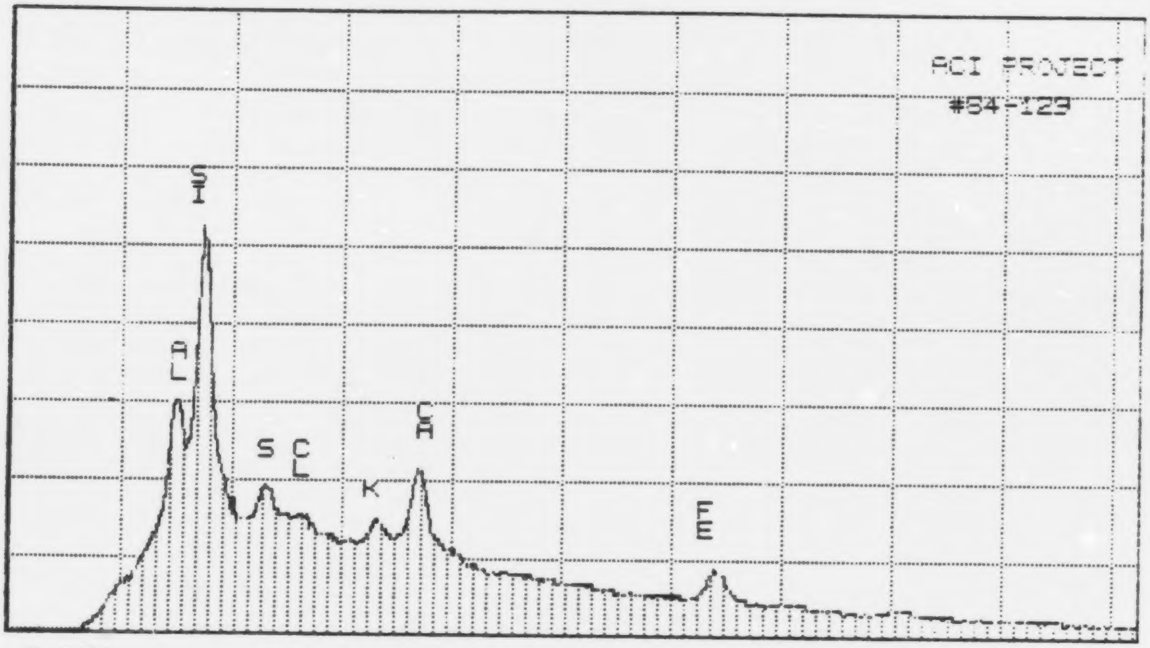


2

0.000 VFS = 8192 10.240

200 DUST 85-175 AREA SCAN 20KV 1000X SUM 4

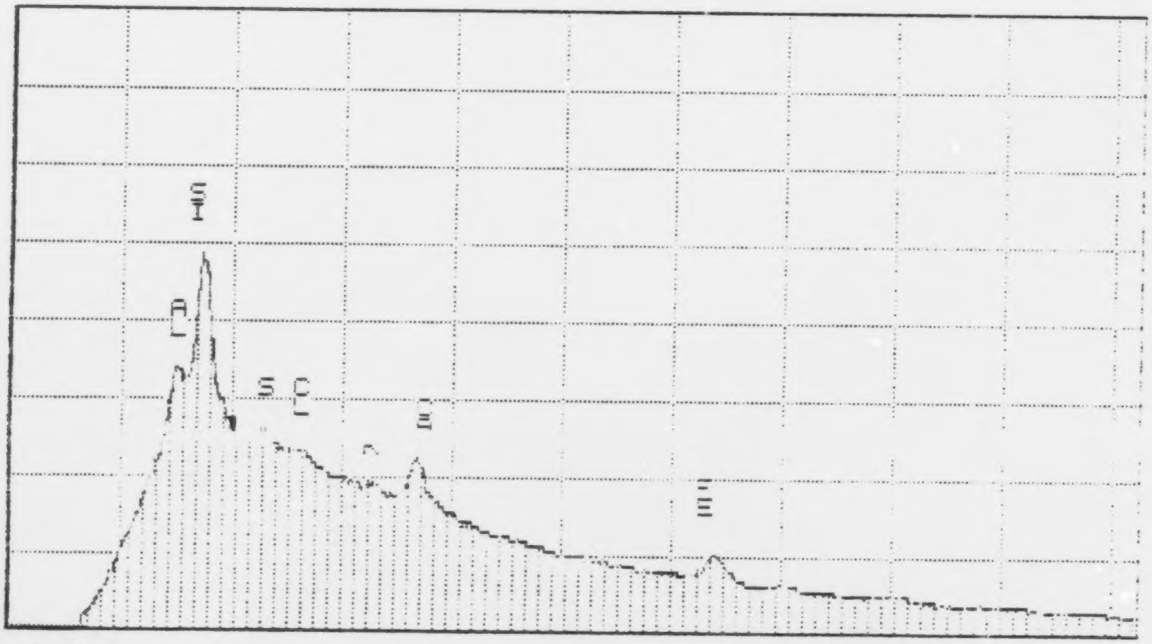
ATLANTIC COAL INSTITUTE TUE 12-MAR-85 16:49
Cursor: 0.000keV = 0



0.000 VFS = 8192 10.240
200 DUST #85-53 20kV AREA RP1 (SUM 4 PTS) 2000X

3

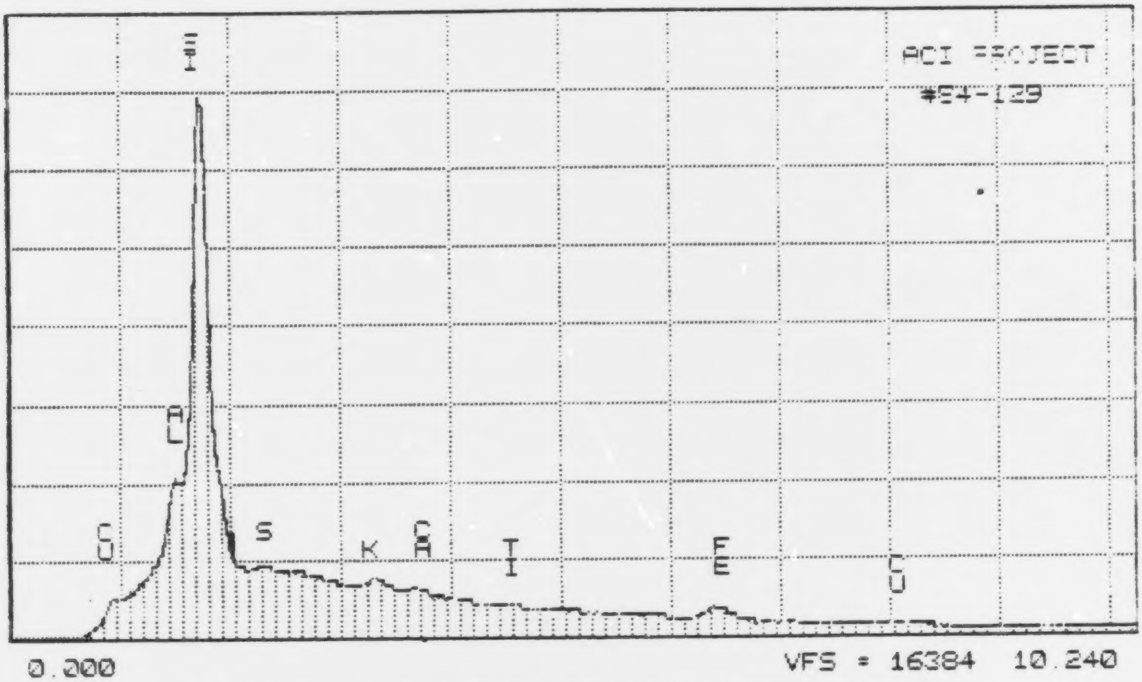
ATLANTIC COAL INSTITUTE WED 23-APR-85 22:33
Cursor: 0.000keV = 0



0.000 VFS = 8192 10.240
200 DUST #85-132 AREA SCAN GENERAL SAMPLE NUCLEP

4

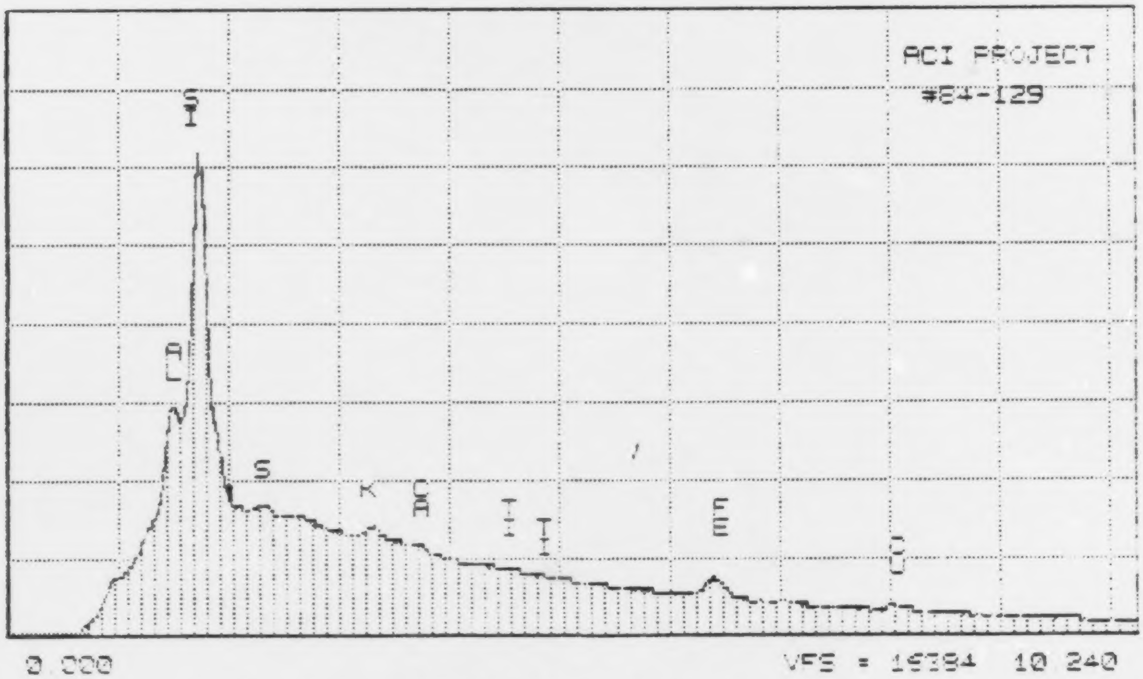
ATLANTIC COAL INSTITUTE   WED 27-MAR-85 15:30
Cursor: 0.000keV = 0



200   DUST 85-39 AREA SCAN RP1 20KV 1000X SUM 4

5

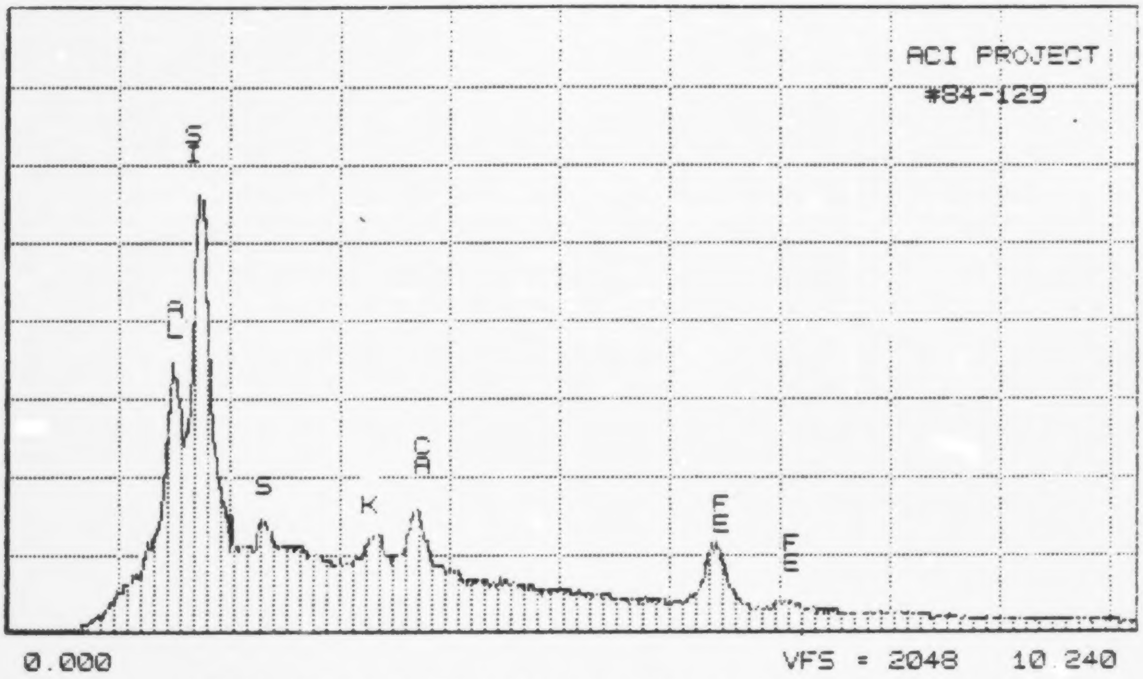
ATLANTIC COAL INSTITUTE   WED 27-MAR-85 15:34
Cursor: 0.000keV = 0



200   DUST 85-9 AREA SCAN RP1 20KV 1000X SUM 4

6

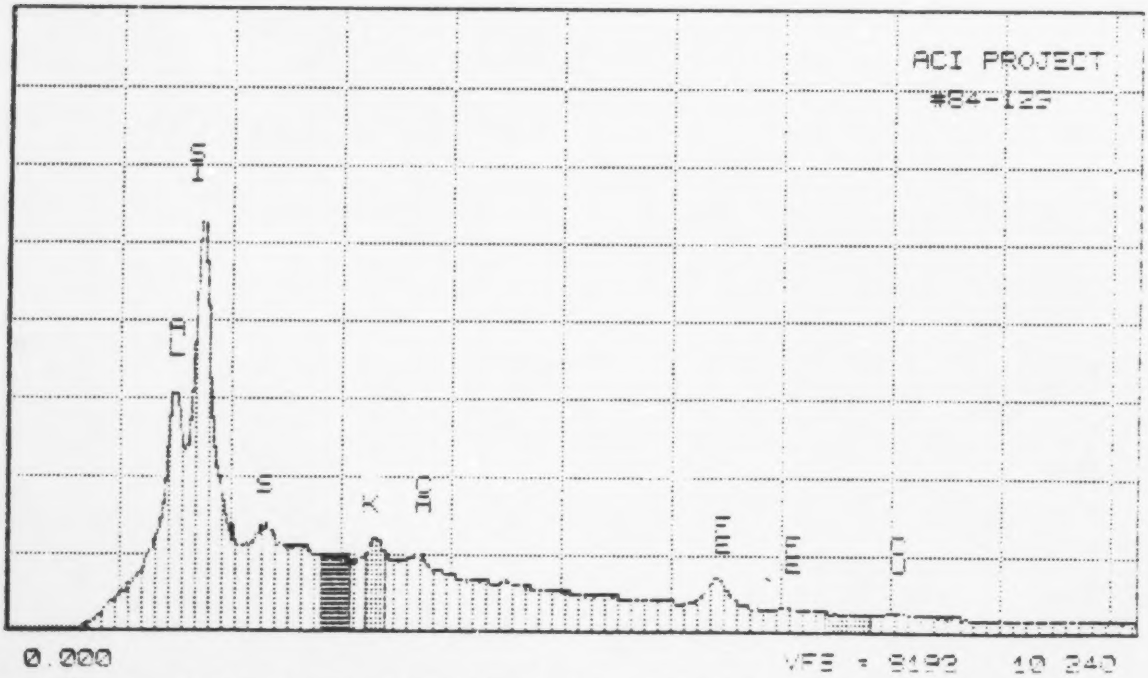
ATLANTIC COAL INSTITUTE FRI 22-MAR-85 09:59
Cursor: 0.000keV = 0



200 DUST #85-36 AREA SCAN RP1 20 KV 750K

7

ATLANTIC COAL INSTITUTE SAT 23-MAR-85 [illegible]
Cursor: 0 000keV = 0 ROI (0) 2.620: 3 070=-442

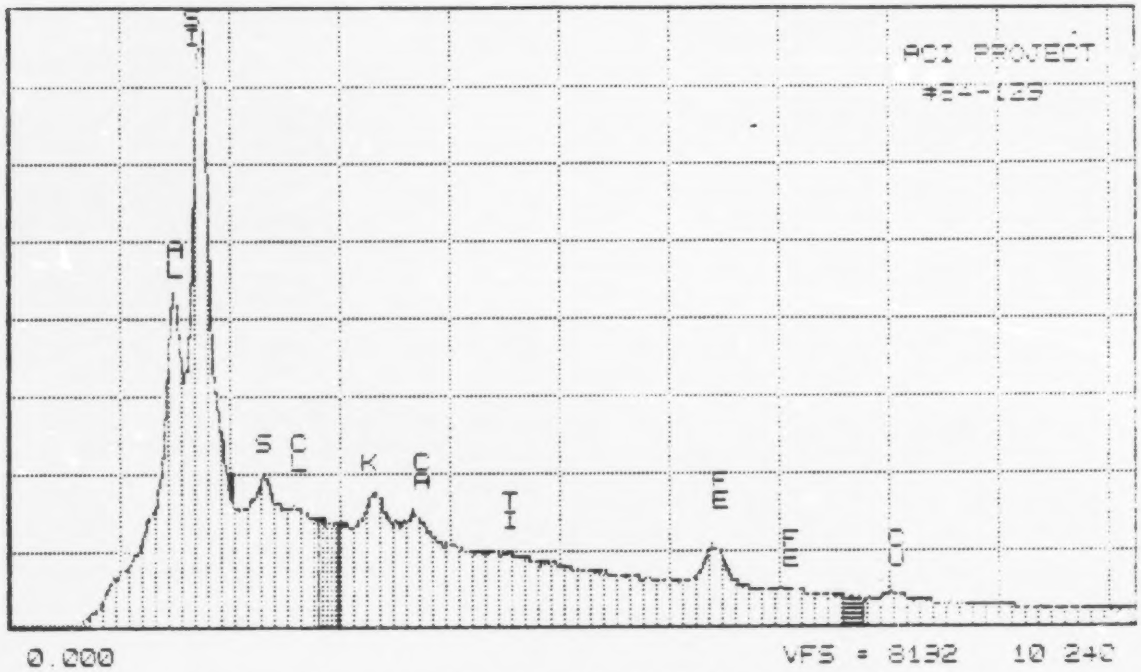


200 DUST 85-75 AREA SCAN RP1 1KX 20KV (SUM 4 [illegible]

8

ATLANTIC COAL INSTITUTE MON 01-APR-85 15:24

Cursor: 0.000keV = 0 ROI (1) 7.580: 7.760=-239

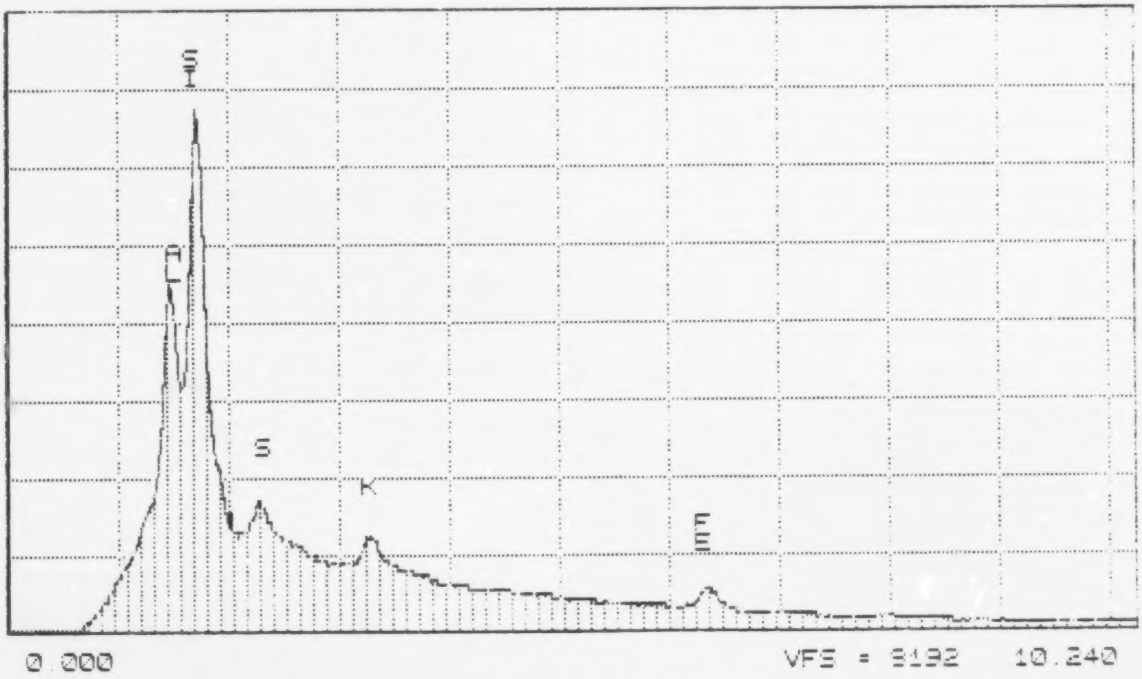


200 DUSTS #85-98 AREA RP1 20KV (SUM 4 PTS) 2000X

9

TN-5500 THU 31-JAN-85 10:23

Cursor: 0.000keV = 0

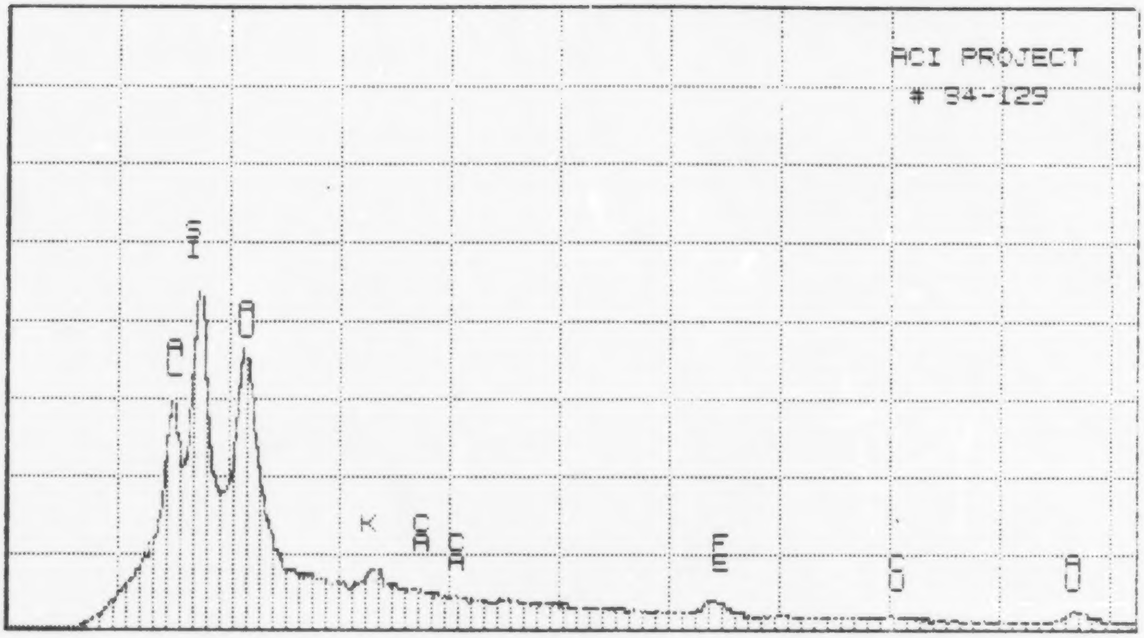


200 85-15 DUST AREA SCAN #0131-01

10

ATLANTIC COAL INSTITUTE MON 18-FEB-95 09:49

Cursor: 0.000keV = 0



0.000 VFS = 4096 10.240

100 DUST CBCRL #85-15 RAS-EXP 500X (2)

11

```
SEMI-QUANTITATIVE ANALYSIS: DUSTS COMPOSITE(SHEARER) AREA SCAN NUCLEPORE    1
EL    NORM. K-RATIO

AL-K 0.17449 +- 0.00079
SI-K 0.30584 +- 0.00100
S -K 0.09859 +- 0.00057
FE-K 0.21938 +- 0.00153
CA-K 0.08897 +- 0.00064
K -K 0.05579 +- 0.00049
CL-K 0.00694 +- 0.00016
TI-K 0.01[illegible] +- 0.000[illegible]
CU-K 0.0[illegible] +- 0.000[illegible]

ZAF CORRECTION        20.00 KV        30.00 Degs

No. of Iterations  3
----      K       [Z]      [A]      [F]    [ZAF]  ATOM.%  WT.%
AL-K   0.174   0.965   1.636   0.983   1.553   22.72   18.44  *
SI-K   0.305   0.941   1.810   0.995   1.697   41.96   35.32  *
S -K   0.090   0.964   1.772   0.995   1.702   10.91   10.50  *
FE-K   0.219   1.143   1.036   0.994   1.178   10.45   17.59
CA-K   0.088   1.006   1.237   0.993   1.238    6.23    7.49
K -K   0.055   1.022   1.295   0.987   1.308    4.24    4.97
CL-K   0.006   1.003   1.635   0.992   1.628    0.73    0.77  *
TI-K   0.013   1.118   1.150   0.985   1.267    0.83    1.19
CU-K   0.044   1.196   1.035   1.000   1.238    1.93    3.72
 * - High Absorbance

SEMI-QUANTITATIVE ANALYSIS: DUST 85-175 AREA SCAN SHEARER NUCLEPORE    2
EL    NORM. K-RATIO

AL-K 0.11464 +- 0.00137
SI-K 0.42717 +- 0.00252
S -K 0.10278 +- 0.00130
FE-K 0.19424 +- 0.00307
CA-K 0.01452 +- 0.00055
K -K 0.02192 +- 0.00066
CL-K 0.04028 +- 0.00084
TI-K-0.00000 +- 0.00000
CU-K 0.08442 +- 0.00266

ZAF CORRECTION        20.00 KV        30.00 Degs

No. of Iterations  0
----      K       [Z]      [A]      [F]    [ZAF]  ATOM.%  WT.%
AL-K   0.114   0.969   1.636   0.977   1.549   14.65   12.05  *
SI-K   0.427   0.945   1.654   0.995   1.556   52.86   45.11  *
S -K   0.102   0.968   1.836   0.996   1.772   12.67   12.36  *
FE-K   0.194   1.147   1.029   0.988   1.167    9.02   15.39
CA-K   0.014   1.010   1.262   0.995   1.270    1.03    1.25
K -K   0.021   1.027   1.359   0.995   1.390    1.74    2.07
CL-K   0.040   1.008   1.710   0.997   1.720    4.41    4.70  *
TI-K   0.000   1.124   1.132   0.985   1.254    0.00    0.00 G
CU-K   0.084   1.202   1.027   1.000   1.236    3.63    7.08
 * - High Absorbance

SEMI-QUANTITATIVE ANALYSIS: DUST #85-53 AREA SCAN SHEARER MILLIPORE    3
EL    NORM. K-RATIO

AL-K 0.12729 +- 0.00108
SI-K 0.33838 +- 0.00168
S -K 0.05836 +- 0.00073
FE-K 0.16651 +- 0.00213
CA-K 0.17846 +- 0.00146
K -K 0.04255 +- 0.00069
CL-K 0.02895 +- 0.00053
TI-K-0.00000 +- 0.00000
CU-K 0.05946 +- 0.00167

ZAF CORRECTION        20.00 KV        30.00 Degs

No. of Iterations  3
----      K       [Z]      [A]      [F]    [ZAF]  ATOM.%  WT.%
AL-K   0.127   0.966   1.652   0.981   1.567   17.33   13.96  *
SI-K   0.338   0.943   1.702   0.995   1.599   45.32   37.86  *
S -K   0.058   0.966   1.756   0.993   1.685    7.21    6.88  *
FE-K   0.166   1.144   1.043   0.992   1.185    8.27   13.82
CA-K   0.178   1.007   1.221   0.995   1.225   12.82   15.30
K -K   0.042   1.024   1.283   0.979   1.287    3.30    3.83
CL-K   0.028   1.005   1.576   0.990   1.570    3.05    3.18  *
TI-K   0.000   1.117   1.183   0.988   1.308    0.00    0.00 G
CU-K   0.059   1.199   1.033   1.000   1.239    2.70    5.16
 * - High Absorbance
```

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SEMI-QUANTITATIVE ANALYSIS: DUST #85-132 AREA SCAN GENERAL SAMPLE NUCLEP   4
EL    NORM. K-RATIO

AL-K 0.04690 +- 0.00089
SI-K 0.36750 +- 0.002[illegible]
S -K 0.03373 +- 0.00076
FE-K 0.22603 +- 0.00337
CA-K 0.15902 +- 0.00187
K -K-0.00000 +- 0.00000
CL-K 0.03132 +- 0.00075
TI-K-0.00000 +- 0.00000
CU-K 0.13546 +- 0.00343

ZAF CORRECTION        20.00 KV        30.00 Degs

No. of Iterations  4
----     K      [Z]     [A]     [F]    [ZAF]  ATOM.%  WT.%
AL-K  0.046  0.948  1.930  0.981  1.798   8.05   6.12  *
SI-K  0.367  0.926  1.700  0.996  1.570  53.09  41.83  *
S -K  0.033  0.948  1.807  0.994  1.704   4.63   4.17  *
FE-K  0.226  1.125  1.036  0.982  1.145  11.92  18.78
CA-K  0.159  0.990  1.197  0.993  1.178  12.07  13.58
K -K  0.000  0.994  1.269  0.975  1.231   0.00   0.00 G
CL-K  0.031  0.987  1.582  0.992  1.551   3.50   3.52  *
TI-K  0.000  1.088  1.155  0.980  1.232   0.00   0.00 G
CU-K  0.135  1.179  1.036  1.000  1.221   6.66  12.00
 * - High Absorbance

SEMI-QUANTITATIVE ANALYSIS: DUST 85-39 AREA SCAN BRUSHER MILLIPORE        5
EL    NORM. K-RATIO

AL-K 0.10388 +- 0.00077
SI-K 0.72064 +- 0.00194
S -K 0.02358 +- 0.00037
FE-K 0.06822 +- 0.00107
CA-K 0.00182 +- 0.00011
K -K 0.00914 +- 0.00025
CL-K 0.01134 +- 0.00026
TI-K-0.00000 +- 0.00000
CU-K 0.06133 +- 0.00134

ZAF CORRECTION        20.00 KV        30.00 Degs

No. of Iterations 17
----     K      [Z]     [A]     [F]    [ZAF]  ATOM.%  WT.%
AL-K  0.103  0.994  1.369  0.959  1.308  11.11   9.97
SI-K  0.720  0.971  1.422  0.998  1.379  77.31  71.97  *
S -K  0.023  0.999  2.144  0.998  2.141   3.48   3.70  *
FE-K  0.068  1.183  1.026  0.990  1.203   3.23   6.82
CA-K  0.001  1.041  1.250  0.997  1.308   0.13   0.10
K -K  0.009  1.058  1.367  0.998  1.445   0.75   0.97
CL-K  0.011  1.040  1.789  0.999  1.860   1.33   1.55  *
TI-K  0.000  1.158  1.124  0.993  1.294   0.00   0.00 G
CU-K  0.061  1.240  1.012  1.000  1.255   2.65   5.65
 * - High Absorbance

SEMI-QUANTITATIVE ANALYSIS: DUST 85-9 AREA SCAN BRUSHER MILLIPORE         6
EL    NORM. K-RATIO

AL-K 0.11552 +- 0.00085
SI-K 0.56415 +- 0.00179
S -K 0.02325 +- 0.00038
FE-K 0.14468 +- 0.00163
CA-K 0.00103 +- 0.00009
K -K 0.02030 +- 0.00039
CL-K 0.01243 +- 0.00029
TI-K-0.00000 +- 0.00000
CU-K 0.11860 +- 0.00195

ZAF CORRECTION        20.00 KV        30.00 Degs

No. of Iterations 12
----     K      [Z]     [A]     [F]    [ZAF]  ATOM.%  WT.%
AL-K  0.115  0.977  1.581  0.970  1.500  13.99  11.84  *
SI-K  0.564  0.950  1.633  0.998  1.551  68.27  59.93  *
S -K  0.023  0.976  2.053  0.998  2.002   3.17   3.18  *
FE-K  0.144  1.157  1.024  0.982  1.165   6.56  11.52
CA-K  0.001  1.019  1.246  0.995  1.266   0.07   0.09
K -K  0.020  1.035  1.343  0.997  1.387   1.57   1.93
CL-K  0.012  1.016  1.729  0.998  1.755   1.36   1.49  *
TI-K  0.000  1.133  1.118  0.987  1.252   0.00   0.00 G
CU-K  0.118  1.212  1.019  1.000  1.236   4.99  10.02
 * - High Absorbance
```

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SEMI-QUANTITATIVE ANALYSIS: DUST #85-38 AREA SCAN SHOTFIRER MILLIPORE      7
EL    NORM. K-RATIO

AL-K 0.17324 +- 0.00217
SI-K 0.33700 +- 0.00289
S -K 0.04075 +- 0.00106
FE-K 0.25682 +- 0.00455
CA-K 0.09619 +- 0.00185
K -K 0.04735 +- 0.00126
CL-K 0.01205 +- 0.00059
TI-K 0.00260 +- 0.00035
CU-K 0.03395 +- 0.00218

ZAF CORRECTION       20.00 KV       30.00 Degs

No. of Iterations  4
----      K       [Z]      [A]      [F]    [ZAF]  ATOM.%  WT.%
[illegible]  [illegible]  [illegible]  [illegible]  [illegible]  [illegible]  [illegible]  [illegible]  *
[illegible]  [illegible]  [illegible]  [illegible]  [illegible]  [illegible]  [illegible]  [illegible]  *
[illegible]  [illegible]  [illegible]  [illegible]  [illegible]  [illegible]  [illegible]  [illegible]  *
FE-K   0.256   1.142   1.033   0.995   1.1[illegible]   12.22   20.59
CA-K   0.096   1.006   1.223   0.993   1.223    6.67    8.03
K -K   0.047   1.022   1.284   0.986   1.295    3.57    4.18
CL-K   0.012   1.001   1.602   0.993   1.593    1.25    1.31  *
TI-K   0.002   1.105   1.143   0.979   1.237    0.15    0.22
CU-K   0.033   1.195   1.037   1.000   1.240    1.49    2.87
 * - High Absorbance
SEMI-QUANTITATIVE ANALYSIS: DUST 85-76 AREA SCAN SHOTFIRER MILLIPORE       8
EL    NORM. K-RATIO

AL-K 0.20284 +- 0.00143
SI-K 0.44179 +- 0.00201
S -K 0.04743 +- 0.00069
FE-K 0.16121 +- 0.00219
CA-K 0.03269 +- 0.00065
K -K 0.04833 +- 0.00077
CL-K 0.01212 +- 0.00036
TI-K-0.00000 +- 0.00000
CU-K 0.05355 +- 0.00166

ZAF CORRECTION       20.00 KV       30.00 Degs

PT.NO.   2

No. of Iterations  2
----      K       [Z]      [A]      [F]    [ZAF]  ATOM.%  WT.%
AL-K   0.202   0.978   1.499   0.977   1.434   22.60   19.41  *
SI-K   0.441   0.954   1.743   0.997   1.659   55.08   48.88  *
S -K   0.047   0.978   1.934   0.997   1.885    5.88    5.97  *
FE-K   0.161   1.150   1.029   0.992   1.184    7.17   12.73
CA-K   0.032   1.020   1.252   0.995   1.273    2.19    2.70
K -K   0.048   1.037   1.323   0.994   1.364    3.56    4.40
CL-K   0.012   1.018   1.689   0.995   1.712    1.25    1.39  *
TI-K   0.000   1.134   1.134   0.988   1.271    0.00    0.00 G
CU-K   0.053   1.214   1.024   1.000   1.243    2.19    4.44
 * - High Absorbance
SEMI-QUANTITATIVE ANALYSIS: DUSTS #85-98 AREA SCAN JACKMAN MILLIPORE      9
EL    NORM. K-RATIO

AL-K 0.18250 +- 0.00111
SI-K 0.44907 +- 0.00167
S -K 0.03951 +- 0.00052
FE-K 0.16538 +- 0.00182
CA-K 0.03772 +- 0.00058
K -K 0.05162 +- 0.00065
CL-K 0.01165 +- 0.00029
TI-K 0.00541 +- 0.00025
CU-K 0.05711 +- 0.00141

ZAF CORRECTION       20.00 KV       30.00 Degs

PT.NO.   1

No. of Iterations  6
----      K       [Z]      [A]      [F]    [ZAF]  ATOM.%  WT.%
AL-K   0.182   0.976   1.529   0.977   1.459   21.11   17.92  *
SI-K   0.449   0.952   1.724   0.997   1.639   56.22   49.50  *
S -K   0.039   0.975   1.927   0.996   1.873    4.95    4.98  *
FE-K   0.165   1.155   1.030   0.991   1.181    7.47   13.15
CA-K   0.037   1.016   1.248   0.995   1.262    2.55    3.21
K -K   0.051   1.034   1.315   0.993   1.351    3.83    4.69
CL-K   0.011   1.013   1.666   0.995   1.680    1.20    1.32  *
TI-K   0.005   1.129   1.134   0.987   1.265    0.31    0.46
CU-K   0.057   1.208   1.026   1.000   1.240    2.37    4.77
 * - High Absorbance
```

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SEMI-QUANTITATIVE ANALYSIS: DUST 85-15 AREA SCAN CARBON COATED            10
EL    NORM. K-RATIO

AL-K 0.28978 +- 0.00153
SI-K 0.46836 +- 0.00186
S -K 0.07505 +- 0.00078
FE-K 0.07222 +- 0.00132
CA-K-0.00000 +- 0.00000
K -K 0.04548 +- 0.00067
CL-K 0.01931 +- 0.00041
TI-K-0.00000 +- 0.00000
CU-K 0.02977 +- 0.00111

ZAF CORRECTION         20.00 KV        30.00 Degs

No. of Iterations  0
----      K      [Z]     [A]     [F]    [ZAF]  ATOM.%  WT.%
AL-K  0.289  0.995  1.282  0.973  1.242  26.52  23.96
SI-K  0.468  0.970  1.704  0.996  1.649  54.86  51.40  *
S -K  0.075  0.994  1.955  0.997  1.940   9.05   9.69  *
FE-K  0.072  1.177  1.029  0.995  1.207   3.10   5.80
CA-K  0.000  1.037  1.279  0.998  1.325   0.00   0.00 G
K -K  0.045  1.054  1.361  0.998  1.433   3.33   4.34
CL-K  0.019  1.035  1.754  0.997  1.812   1.99   2.33  *
TI-K  0.000  1.153  1.135  0.994  1.303   0.00   0.00 G
CU-K  0.029  1.233  1.014  1.000  1.251   1.16   2.48
 * - High Absorbance

SEMI-QUANTITATIVE ANALYSIS: DUST 85-15 AREA SCAN GOLD COATED              11
EL    NORM. K-RATIO

AL-K 0.23887 +- 0.00222
SI-K 0.41228 +- 0.00278
S -K 0.17152 +- 0.00109
FE-K 0.08059 +- 0.00222
CA-K-0.00000 +- 0.00000
K -K 0.03382 +- 0.00092
CL-K 0.01213 +- 0.00052
TI-K-0.00000 +- 0.00000
CU-K 0.05076 +- 0.00232

ZAF CORRECTION         20.00 KV        30.00 Degs

No. of Iterations  2
----      K      [Z]     [A]     [F]    [ZAF]  ATOM.%  WT.%
AL-K  0.238  0.990  1.356  0.975  1.310  23.41  20.64
SI-K  0.412  0.965  1.671  0.993  1.604  47.70  43.62  *
S -K  0.171  0.989  1.828  0.998  1.805  19.54  20.43  *
FE-K  0.080  1.172  1.032  0.992  1.200   3.49   6.38
CA-K  0.000  1.032  1.290  0.997  1.338   0.00   0.00 G
K -K  0.033  1.049  1.388  0.998  1.455   2.55   3.25
CL-K  0.012  1.030  1.826  0.998  1.878   1.32   1.50  *
TI-K  0.000  1.140  1.141  0.993  1.302   0.00   0.00 G
CU-K  0.050  1.228  1.016  1.000  1.240   2.00   4.18
 * - High Absorbance
```

6a. PHOTOMICROGRAPHS: COAL DUST PARTICULATES

1. Coal dust particles on glass fibre filter, gold coated. An EDX spectrum was obtained at the crosshairs (EDX #1), but the filter matrix and the particle agglomeration prevent differentiation. x5000, 15kv in Secondary Electron Image (SEI) mode.

2. Coal dust on glass fibre filter. Note the extreme depth of field for such a filter substrate. x10,000, SEI, 15kv.

3. Coal dust particles on PVC filter, gold coated. x15,000, SEI, 15kv. Spot 1 yields an ambiguous EDX spectrum (EDX #3,4) little different from the background. Identification is not possible due to the substrate.

4. ibid: Quartz, x20,000 (EDX #5) Note the diameter of the grain at 1 micron.

5. Coal dust on Nuclepore filter (8.0um) sample #85-22, showing particle charging due to discontinuous carbon coating. x5000, SEI, 15kv. The EDX spectrum (#6) suggests illite clay.

6. Coal dust on Nuclepore #85-22. Grains are agglomerated to give a composite EDX spectrum (#7). x3500, SEI, 15kv.

7. Coal dust on Nuclepore #85-22. Elongate coal maceral fragment (EDX #8), x5000, SEI.

8. ibid. Illite flake x5000 (EDX #9).

9. Coal dust on Nuclepore #85-132(0.8um pores). Gold coated, x3500, 20kv in backscattered electron image mode (BEI). With gold coating the mean Z differences among grains is less distinguished than for carbon coated specimens.

10. ibid. Agglomerated flakes of illite with possibly calcite at crosshairs (EDX #10), x5000, BEI, 20kv.

11. ibid. Pyrite at crosshairs, brighter than adjacent grains with lower mean Z. x5000, BEI (EDX #11).

12. Same field of view as photo #11 showing pyrite in SEI mode. Image is sharper but mean Z is not evident. Pyrite is, therefore, difficult to distinguish. x5000, SEI, 20kv.

13. Coal dust on Nuclepore #85-132, as above. Pyrite octahedra of approximately 1um dimensions. x10,000, BEI, 20kv.

14. Same field of view as photo 11. Grain #1 is a coal maceral (EDX #12) x5000, BEI.

15. Siderite or hematite particle on Nuclepore filter #85-132. The EDX (13) spectrum at crosshairs yields mainly Fe. x15000, SEI.

16. Calcite particle on Nuclepore #85-132. (EDX #14). x15000, SEI, 20kv.

17. Agglomerated grains of calcite on Nuclepore #85-132. Likely stone dust. x15000, SEI.

18. Respirable coal dust on Millipore filter #85-38 (0.8um pores). Carbon coated. This and subsequent photos show considerable beam charging artifacts in the SEI mode, which may contribute to "noisy" spectra (EDX #15). At crosshairs is siderite or iron oxide (hematite). x10,000, SEI, 20kv.

19. ibid. pyrite grain x10,000, EDX #16.

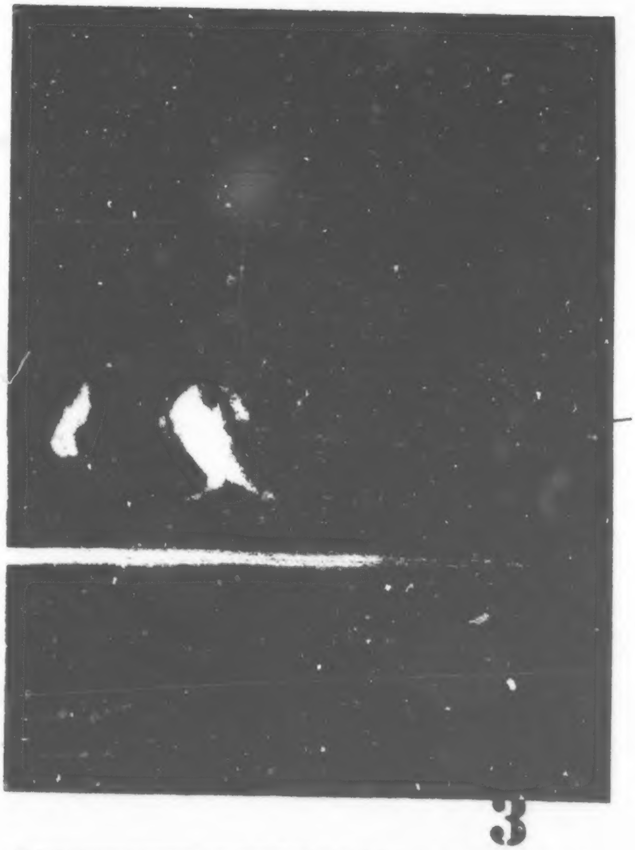
20. ibid. quartz grain x15,000, EDX #17.

21. ibid. Kaolinite grain x10,000, EDX #18. Note that there is no distinctive morphology to differentiate the pyrite, quartz, illite, and kaolinite.

22. Coal dust on Nuclepore #85-132, gold coated: barite grain x35,000, SEI, EDX #19.

1

2



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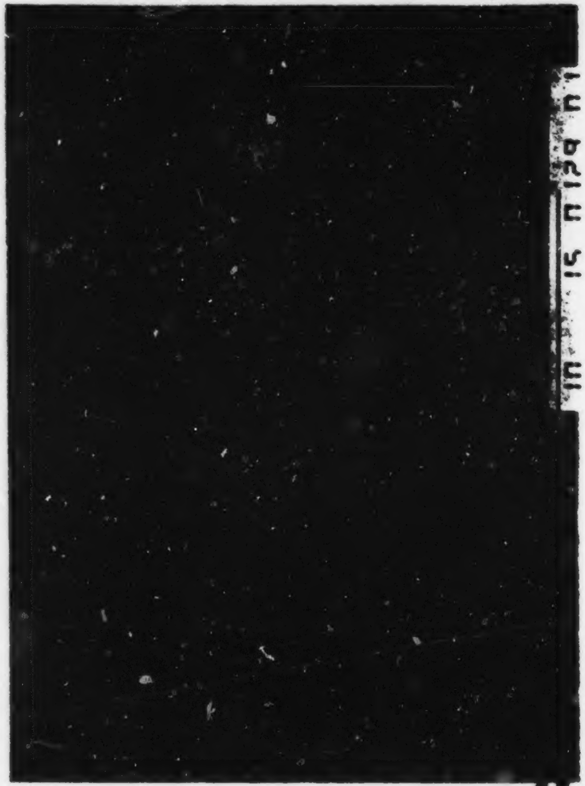

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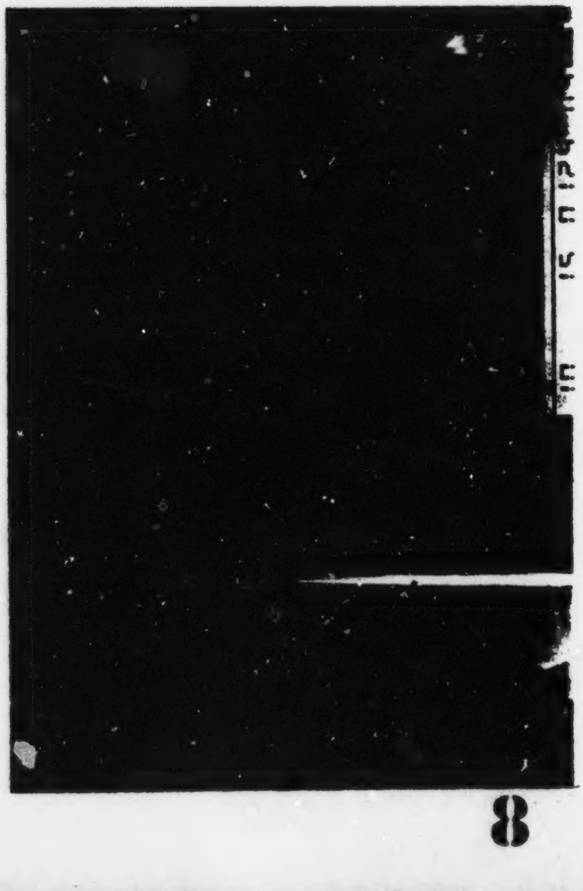

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9

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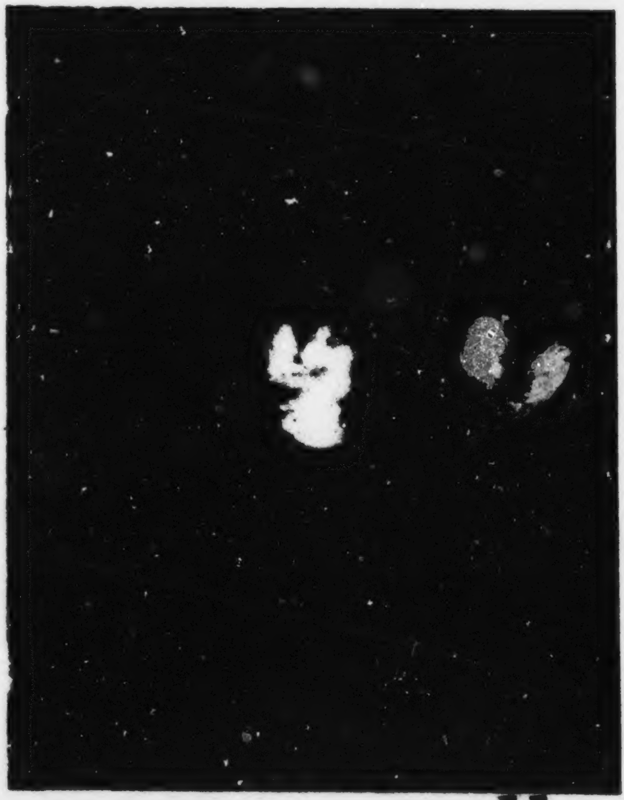

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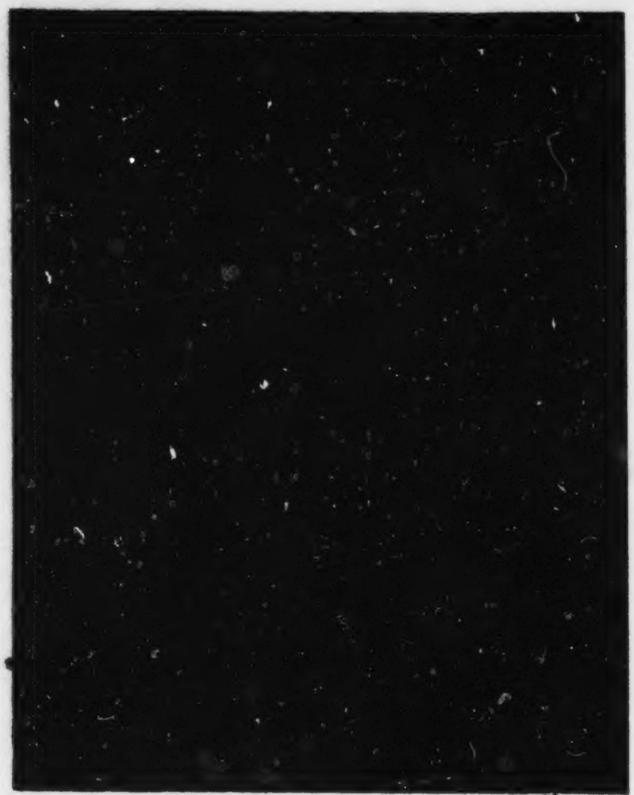

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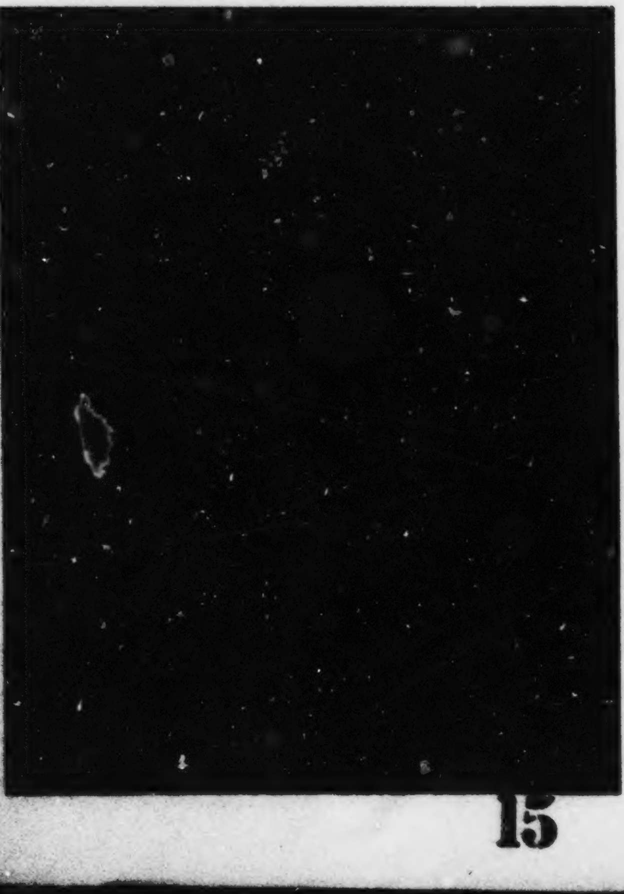

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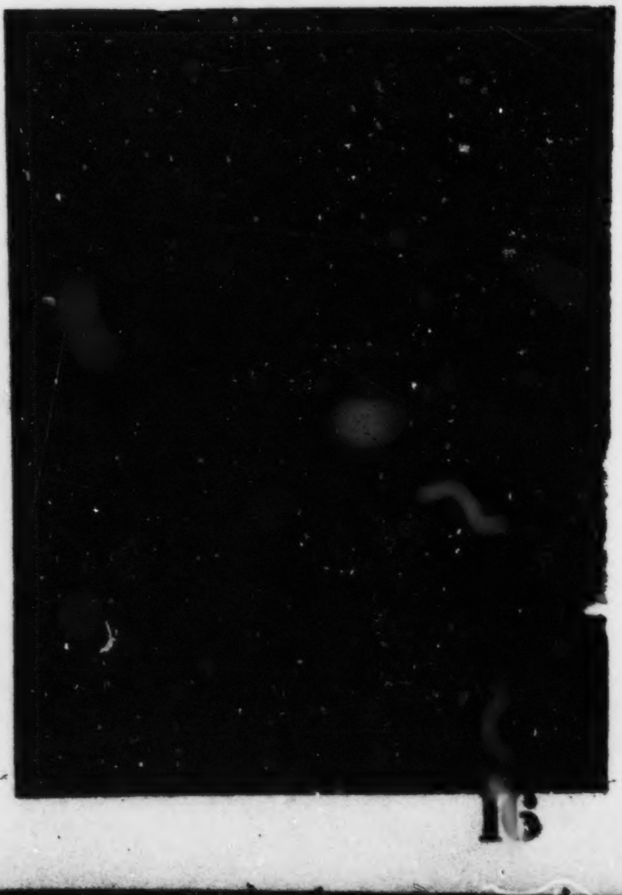

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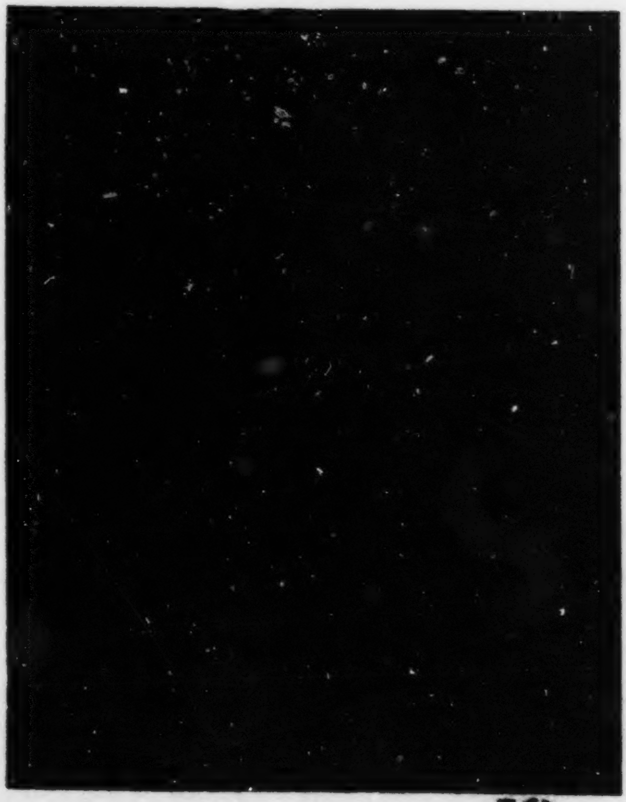
17

18



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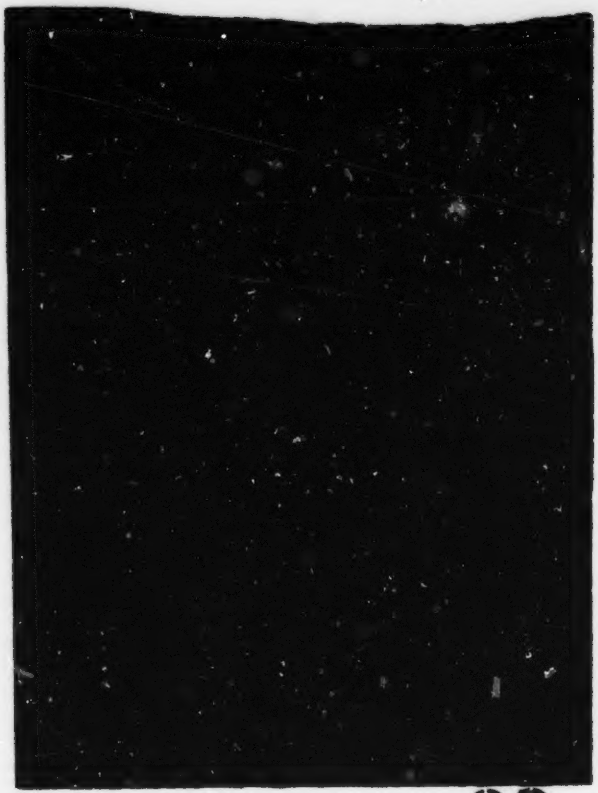

22

6b. EDX SPECTRA: COAL DUST PARTICLES

1. EDX spectrum for a 50 second spot analysis on coal dust particles collected on glass fibre filter material. The particle identity is indistinguishable because of agglomeration, gold coating, and the silica from filter matrix (photo #1), 15kv.

2. EDX spectrum of the glass fibre filter matrix (photo #1), 15kv, spot analysis.

3. EDX spectrum for PVC filter background (photo #3), 200 second counts, 15kv, gold coating.

4. EDX spectrum of coal dust particle on PVC filter (spot 1, photo #3). The dominance of the gold peak (from coating) and chlorine peak (from filter) suggests the particle may be a maceral, contributing minor Si, Al, and Xray continuum.

5. EDX spectrum for quartz (photo #4) at spot #1 based on a 200 second count.

6. EDX spectrum for illite (photo #5) on Nuclepore filter #85-22. The unidentified peak is likely sulphur since the filter was carbon coated.

7. EDX spectrum for agglomerated grains yielding a complex spectrum, likely due to pyrite and chlorite (photo #6).

8. EDX spectrum for coal maceral (photo #7) distinguished by the large biased xray continuum and the strong Si and S peaks. (photo #7). Counts were for 100 seconds in spot mode at 15kv. Note the low vertical full scale.

9. EDX for illite (photo #8)

10. EDX spectrum for coal dusts on Nuclepore filter #85-132, gold coated. These agglomerated flakes (photo #10) are dominantly illite, but the presence of a Ca peak suggests associated calcite from stone dust. Counts for 10 seconds, 20kv.

11. EDX for pyrite (photo #11) at 10 sec. count.

12. EDX for coal maceral (photo #14). Note the suppressed peaks at a VFS similar to spectrum #11. An expanded scale would look like spectrum #8.

13. Hematite or siderite spectrum (photo #15). Note the chlorine peak. This particle is on Nuclepore gold coated, therefore, only the Au peak is artifact.

14. Calcite spectrum (photo #16).

15. EDX for siderite/hematite on Millipore filter #85-38, carbon coated. 50 second counts were taken at 20kv in spot mode (photo #18).

16. EDX for pyrite (photo #19) on filter 85-38.

17. EDX for quartz (photo #20) on filter 85-38.

18. EDX for kaolinite (photo #21) on filter 85-38.

19. EDX for barite on Nuclepore filter 85-132. The triple barite peaks and broad Au-S overlapping peaks are diagnostic for a gold coated sample. Photo #22.

ATLANTIC COAL INSTITUTE MON 21-JAN-85 11:48

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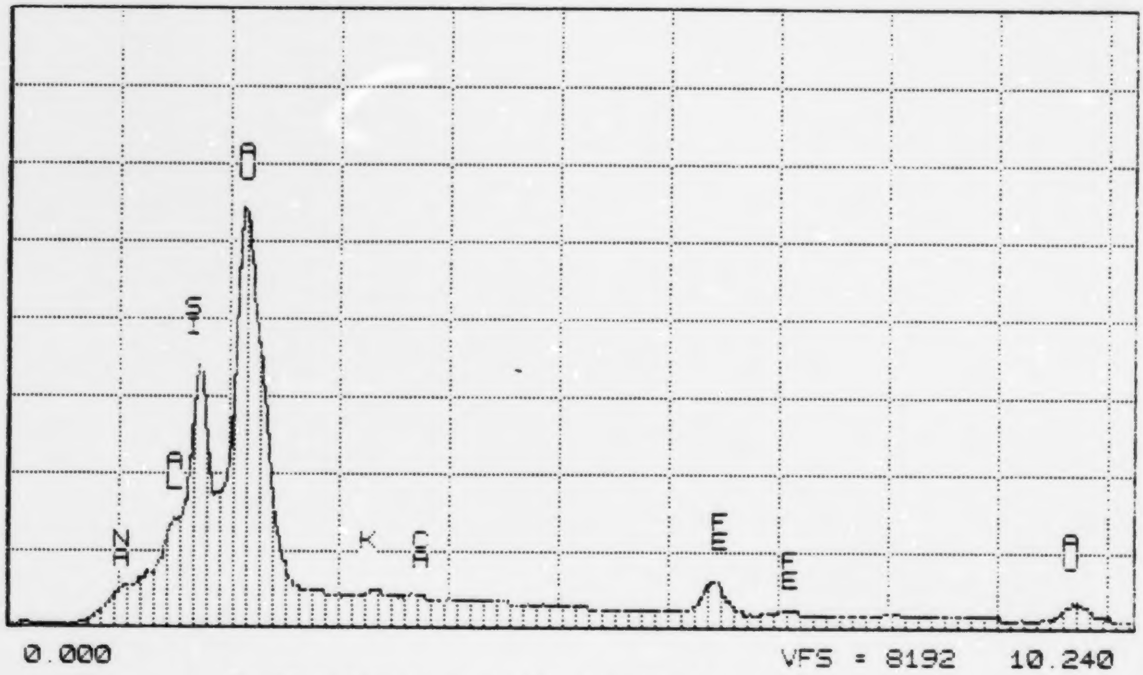

50 DUST PARTICLE,FIBREGLASS,#01210,SPOT

1

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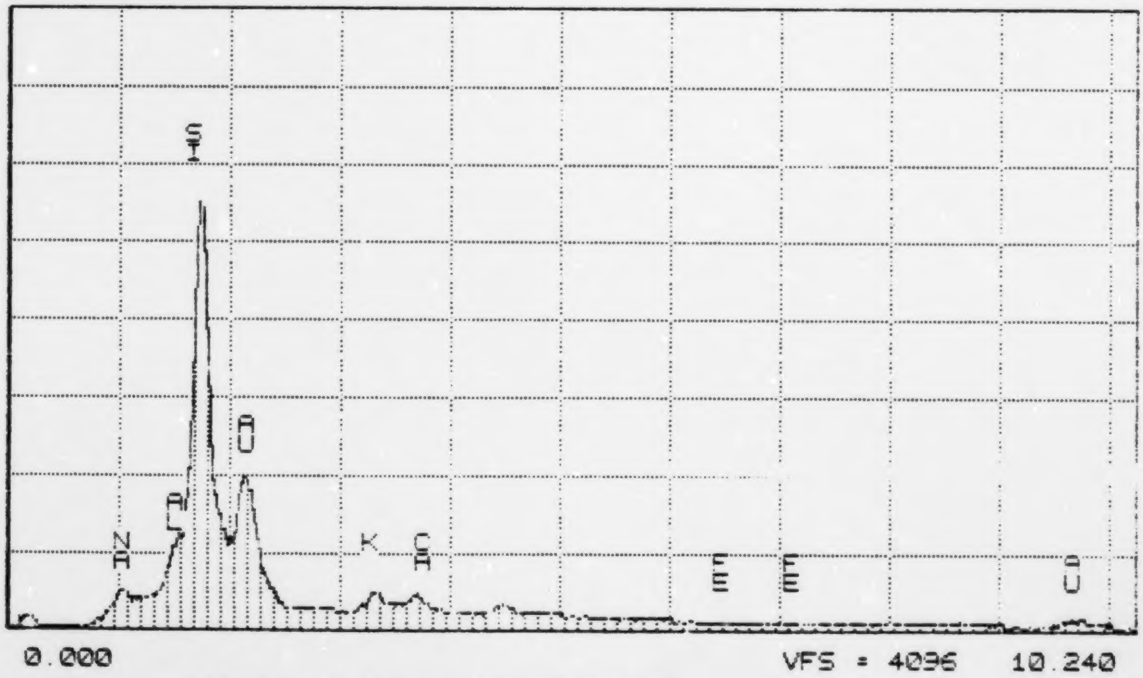

50 FIBREGLAS BACKGROUND RASTER

2

ATLANTIC COAL INSTITUTE THU 17-JAN-85 16:18

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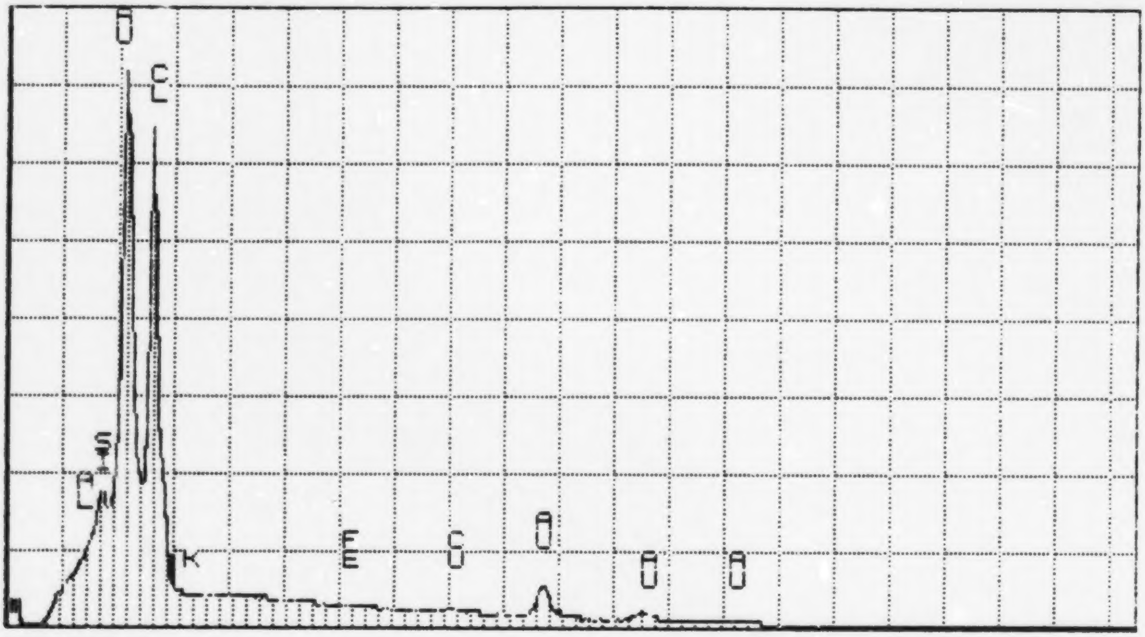

0.000 VFS = 8192 20.480

200 BACKGROUND,PVC FILTER,15KV,PHOTO 011703

3

ATLANTIC COAL INSTITUTE THU 17-JAN-85 [illegible]

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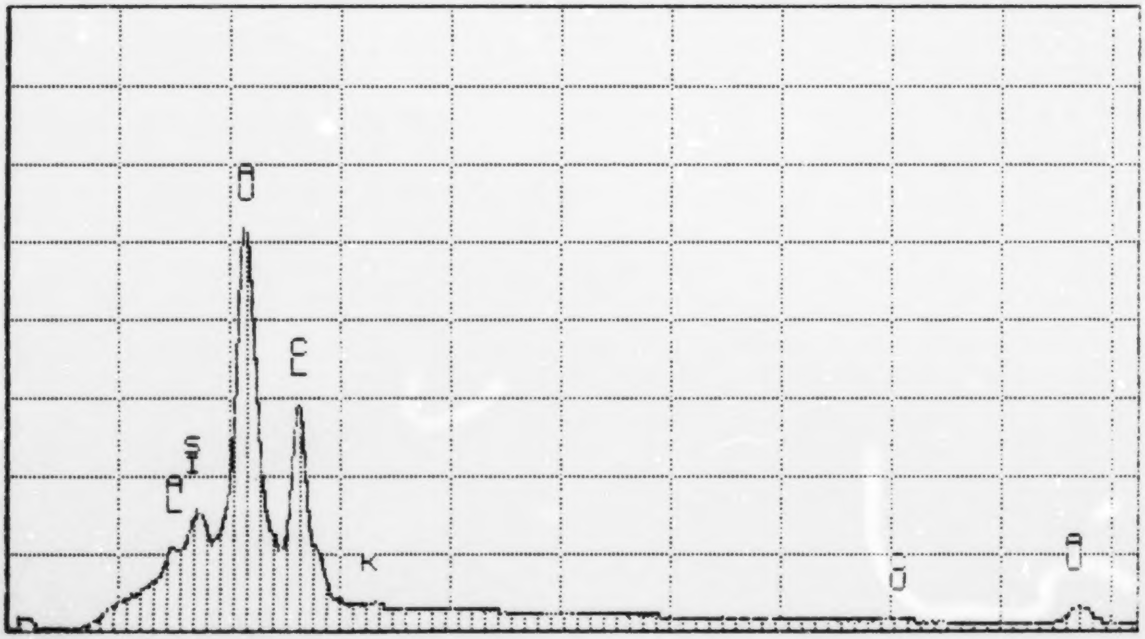

0.000 VFS = 16384 10.240

200 DUST PARTICLE,PVC FILTER,15KV,PHOTO 011702

4

ATLANTIC COAL INSTITUTE THU 17-JAN-85 16:31

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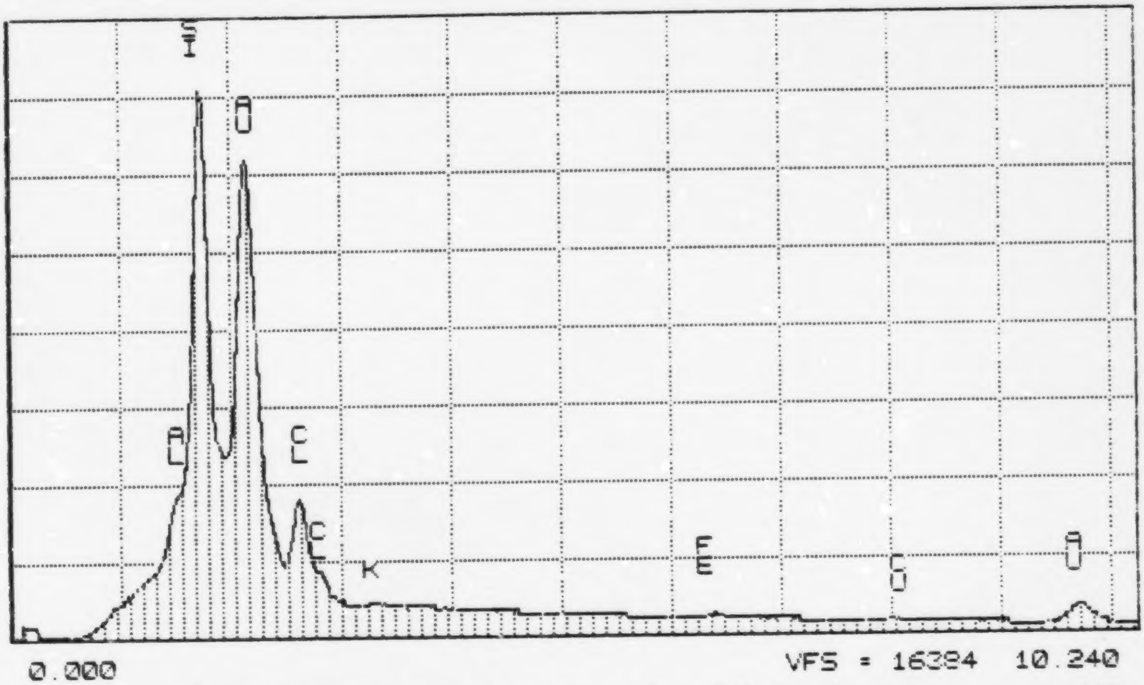

DUST PARTICLE, PVC FILTER, 15KV, #011703 SPOT 200

5

TN-5500 MON 28-JAN-85 12:01

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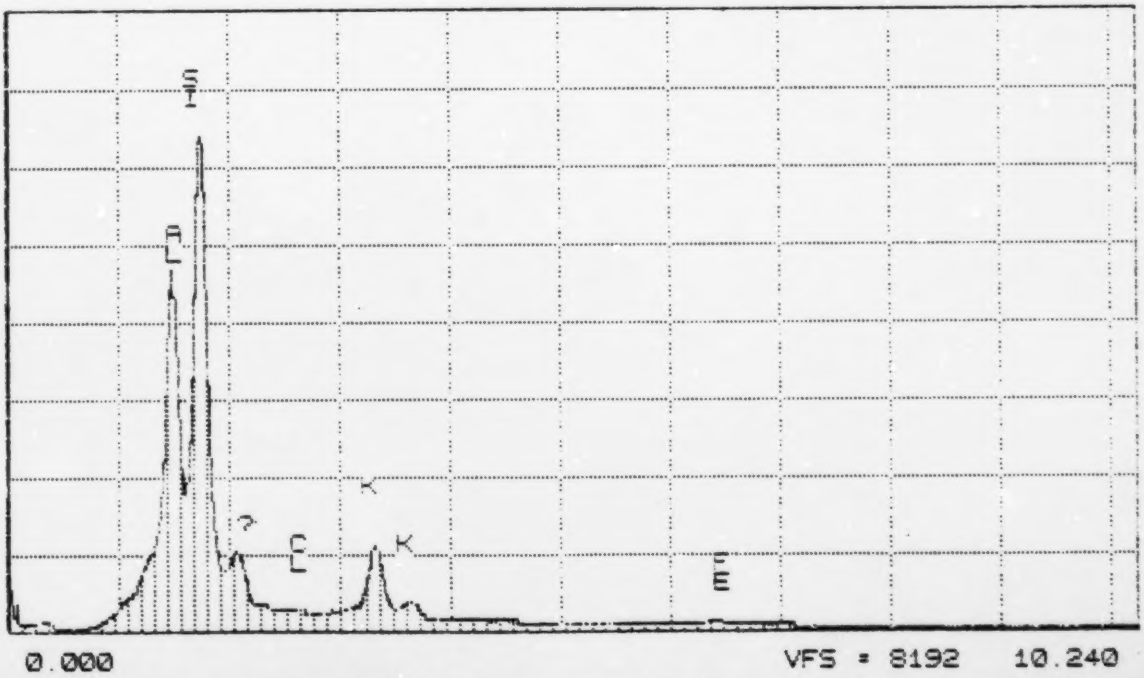
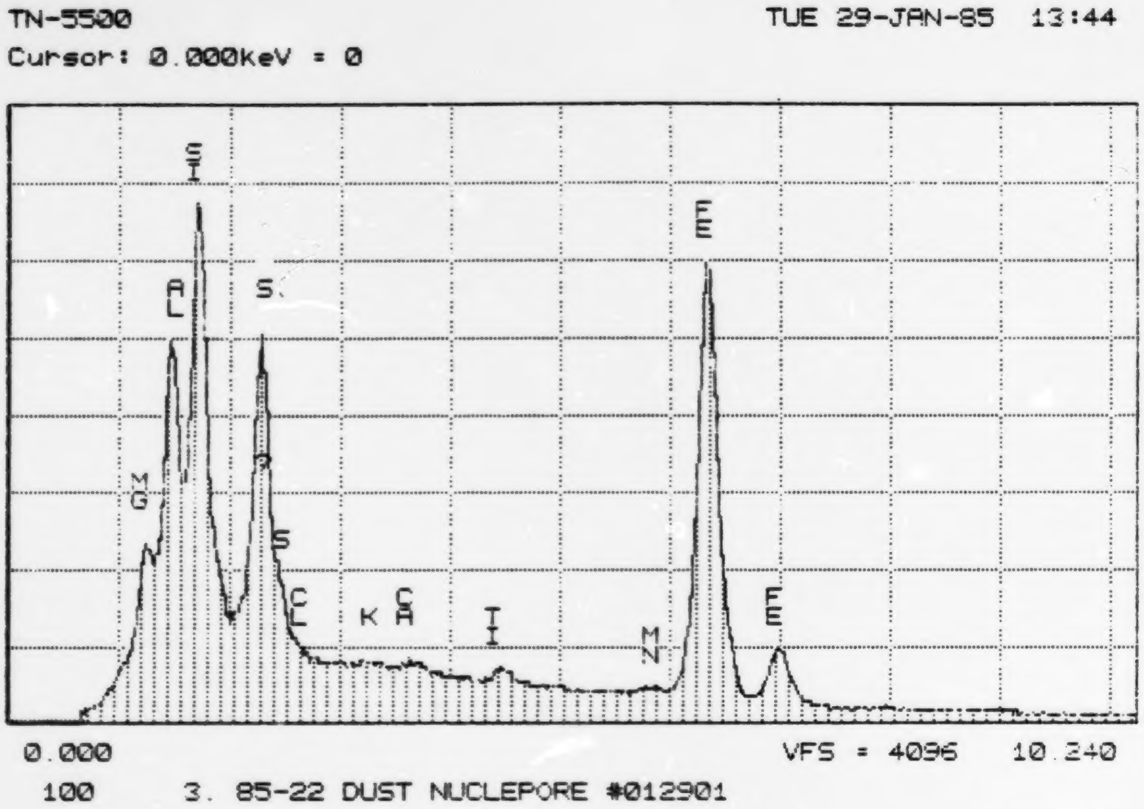


106 1. 85-22 DUST NUCLEPORE #012801

6



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TN-5500                                   TUE 29-JAN-85  13:44
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 100      3. 85-22 DUST NUCLEPORE #012901
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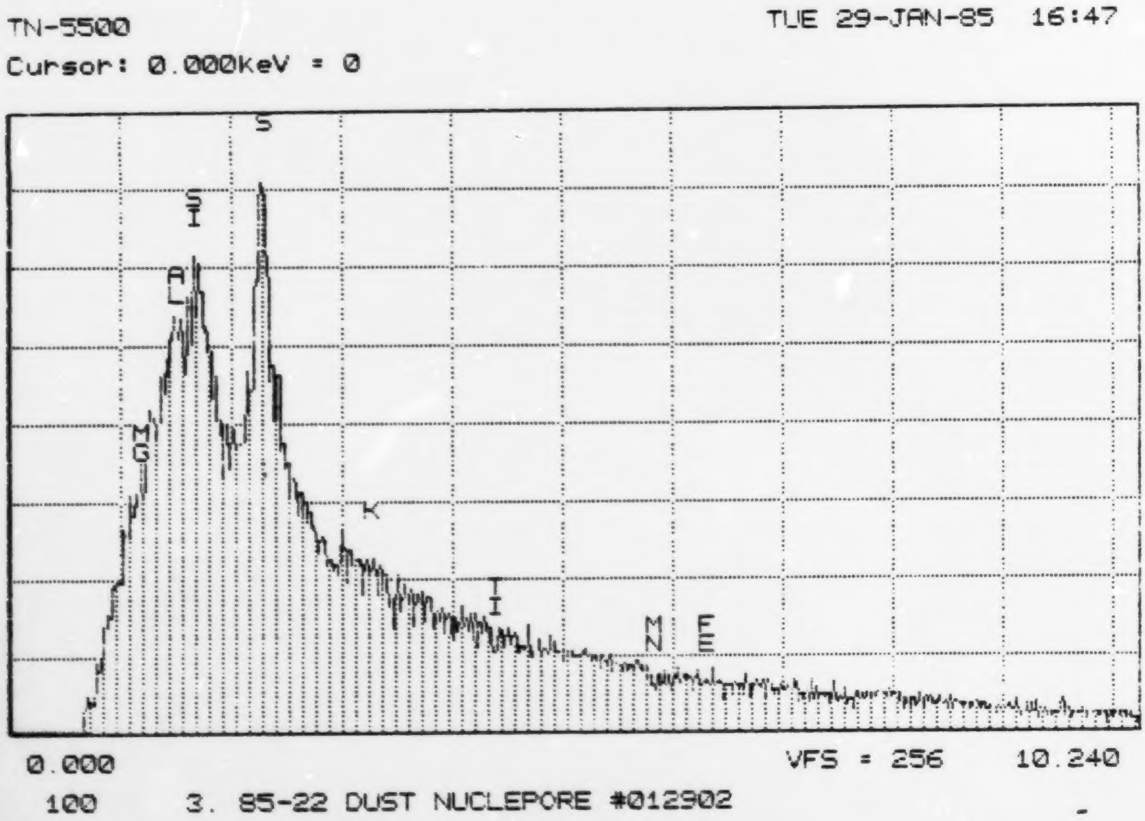
7



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 100      3. 85-22 DUST NUCLEPORE #012902
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8

TN-5500 TUE 29-JAN-85 16:51

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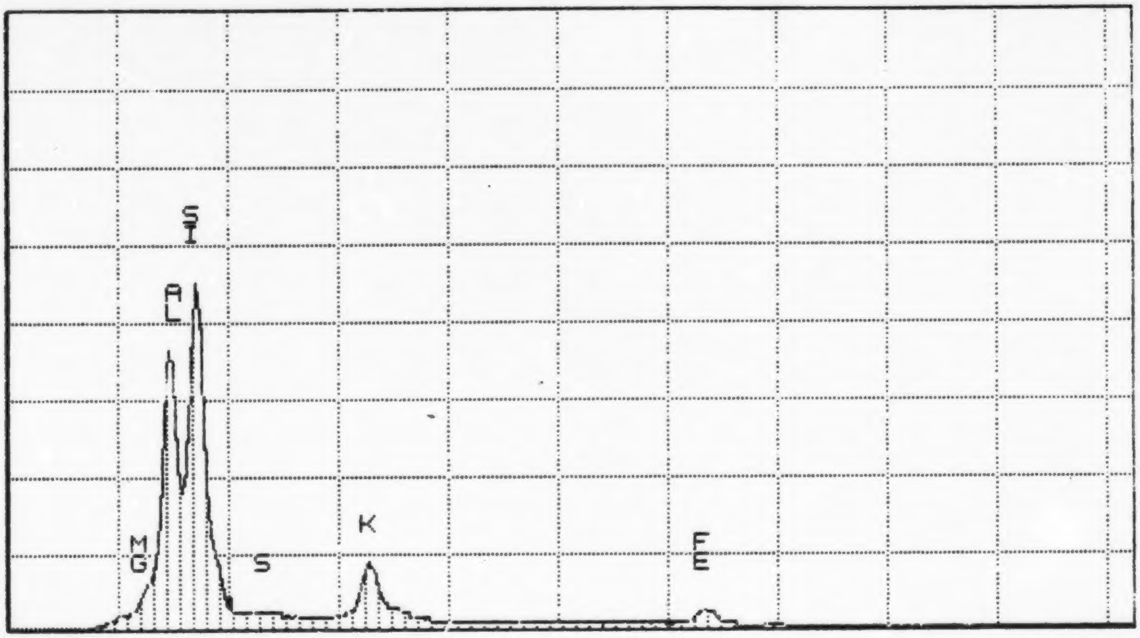

0.000 VFS = 16384 10.240

100 3. 85-22 DUST NUCLEPORE #012905

9

ATLANTIC COAL INSTITUTE TUE 05-MAR-85 11:51

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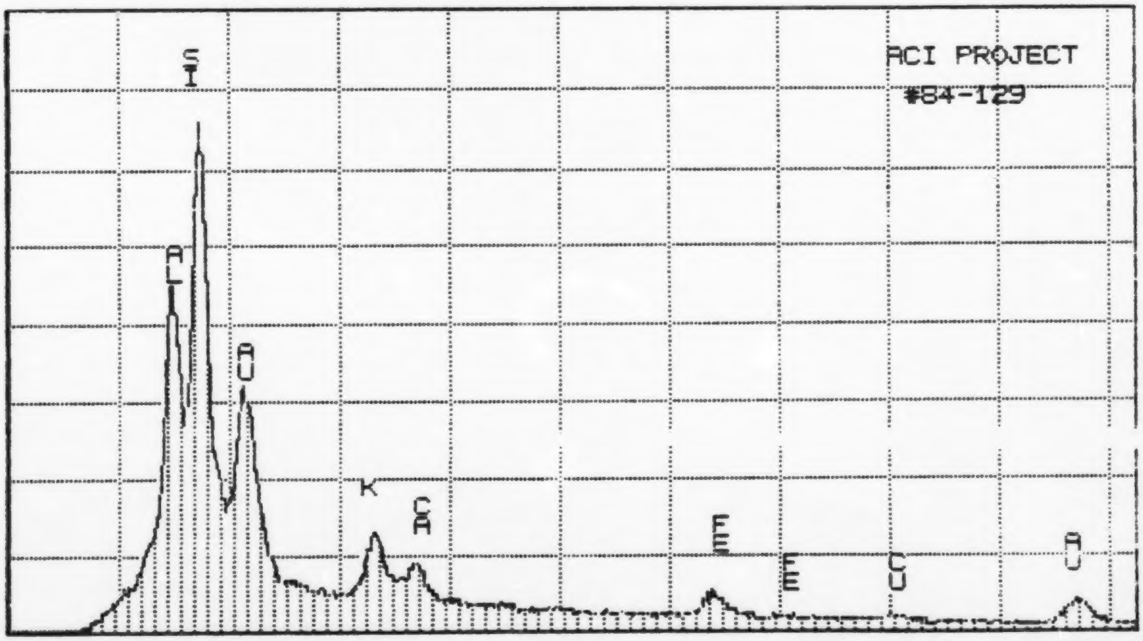

0.000 VFS = 4096 10.240

10 DUST 85-132 MINERAL IDS #0304-04 5000X

10

ATLANTIC COAL INSTITUTE TUE 05-MAR-85 11:53
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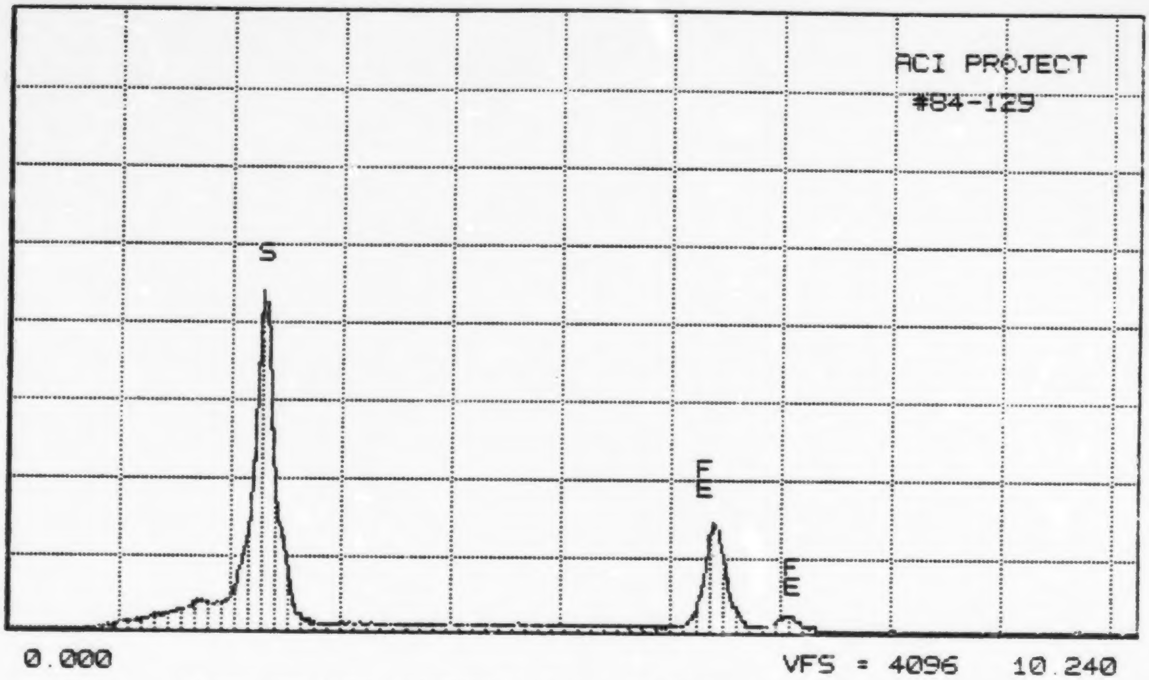

10 DUST 85-132 MINERAL IDS #0305-01 5000X

11

ATLANTIC COAL INSTITUTE TUE 05-MAR-85 11:57
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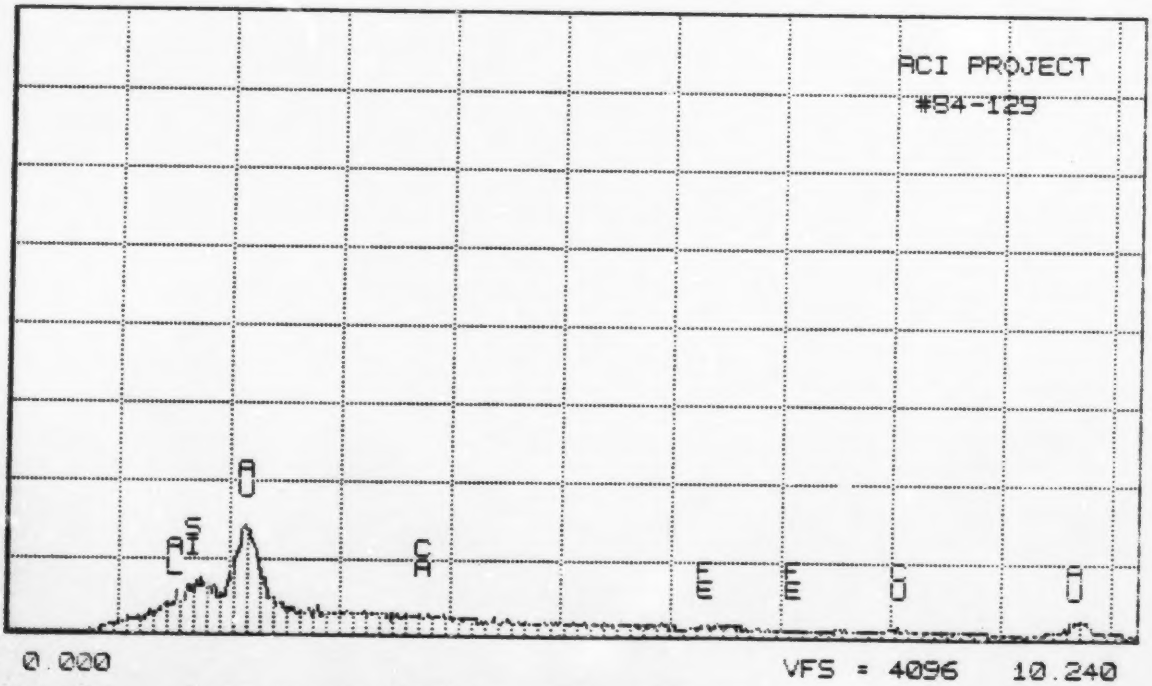

10 DUST 85-132 MINERAL IDS #0305-02

12

ATLANTIC COAL INSTITUTE THU 21-MAR-85 16:30
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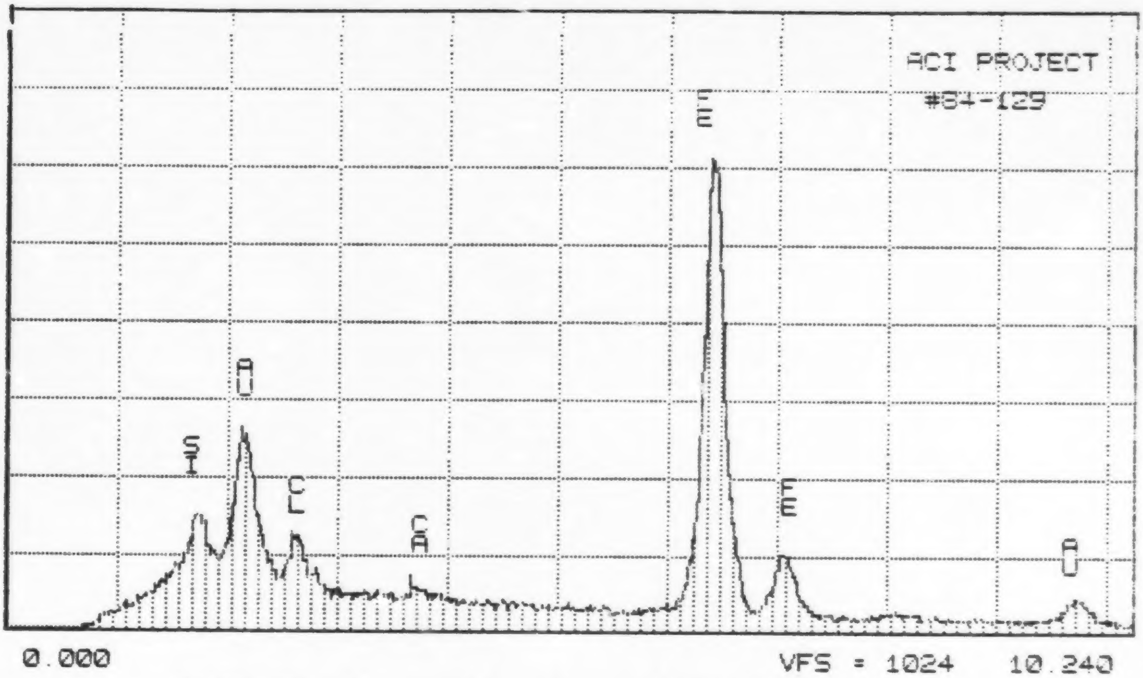

10 DUST 85-132 MINERAL IDS #0305-06 SPOT

13

ATLANTIC COAL INSTITUTE THU 21-MAR-85 16:40
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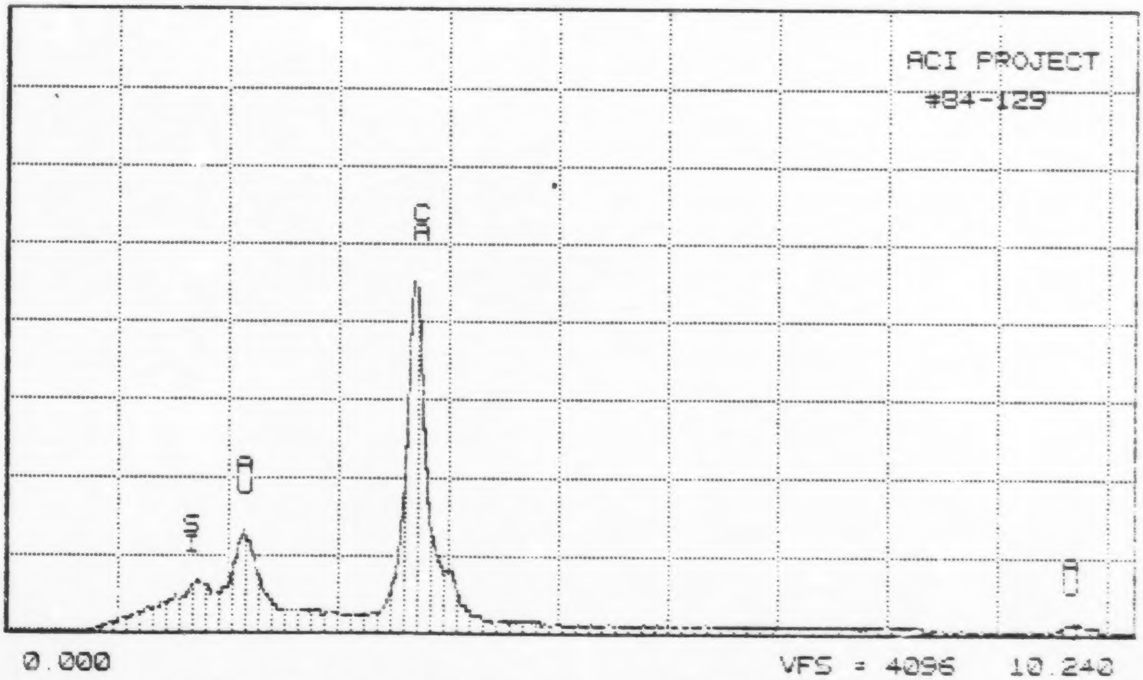

10 DUST 85-132 MINERAL IDS #0305-07 SPOT

14

ATLANTIC COAL INSTITUTE THU 07-MAR-85 16:39

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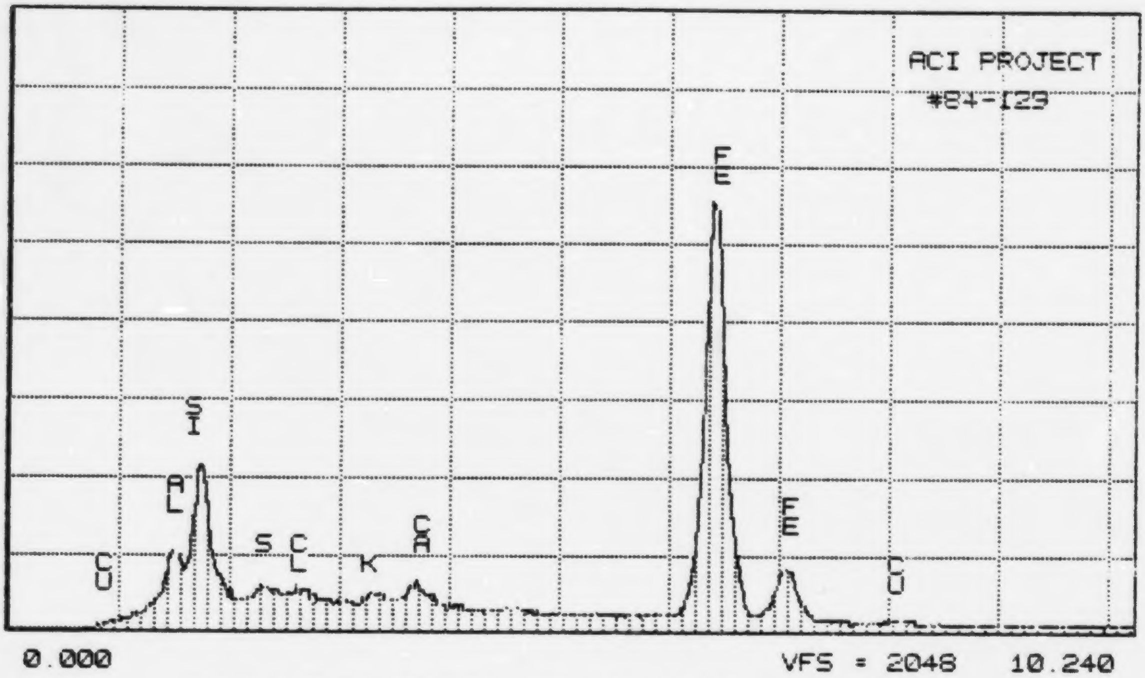

50 DUST #85-38 MINERAL ID 20kV SPOT 0307-01

15

ATLANTIC COAL INSTITUTE THU 07-MAR-85 16:41

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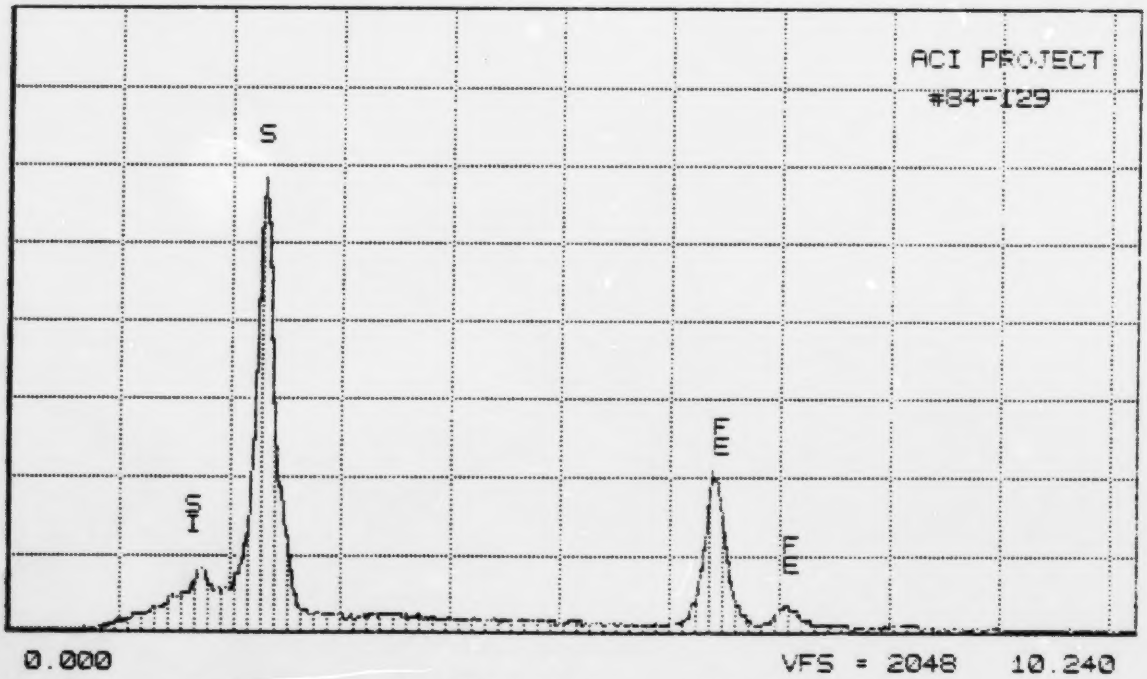

50 DUST #85-38 MINERAL ID 20kV SPOT 0307-02

16

ATLANTIC COAL INSTITUTE THU 07-MAR-85 16:43
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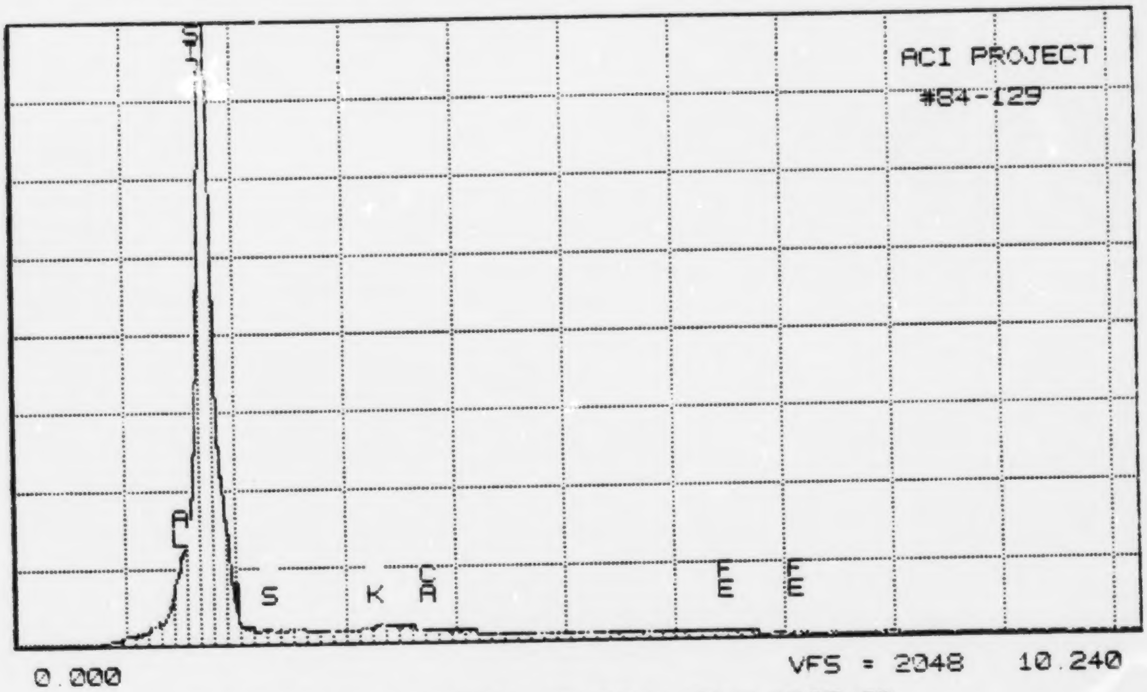

17

25 DUST #85-38 MINERAL ID 20KV SPOT 0307-03

ATLANTIC COAL INSTITUTE THU 07-MAR-85 16:55
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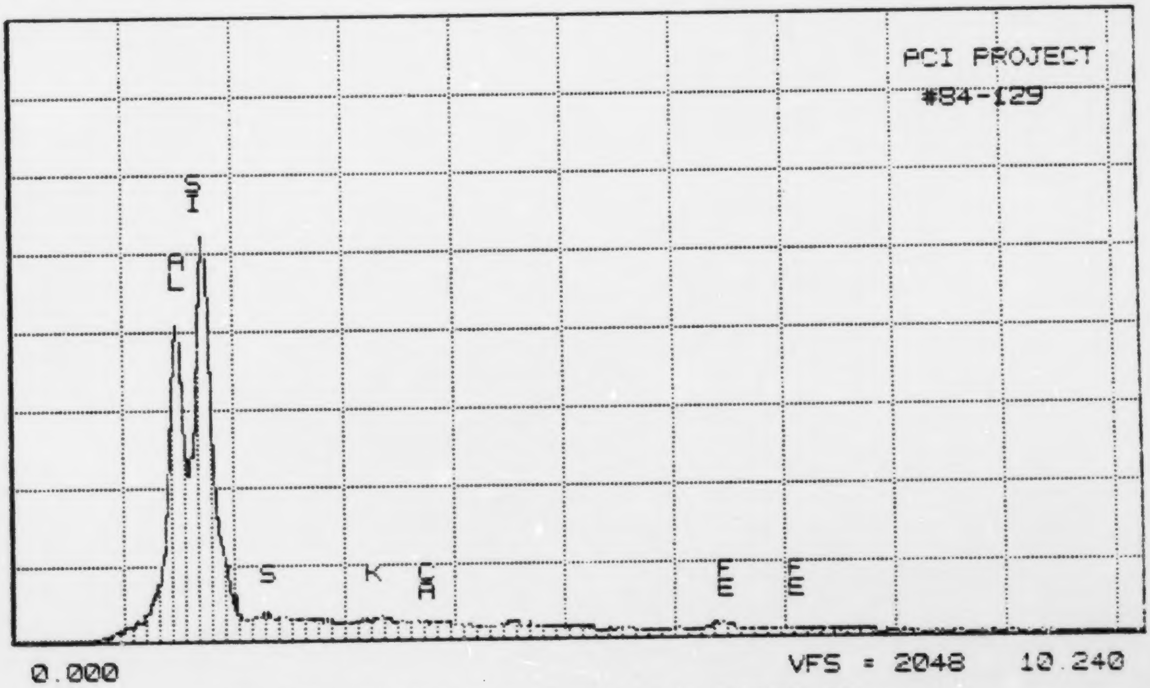

18

10 DUST #85-38 MIN ID 20 KV SPOT P0307-07

ATLANTIC COAL INSTITUTE   WED 10-APR-85 17:11

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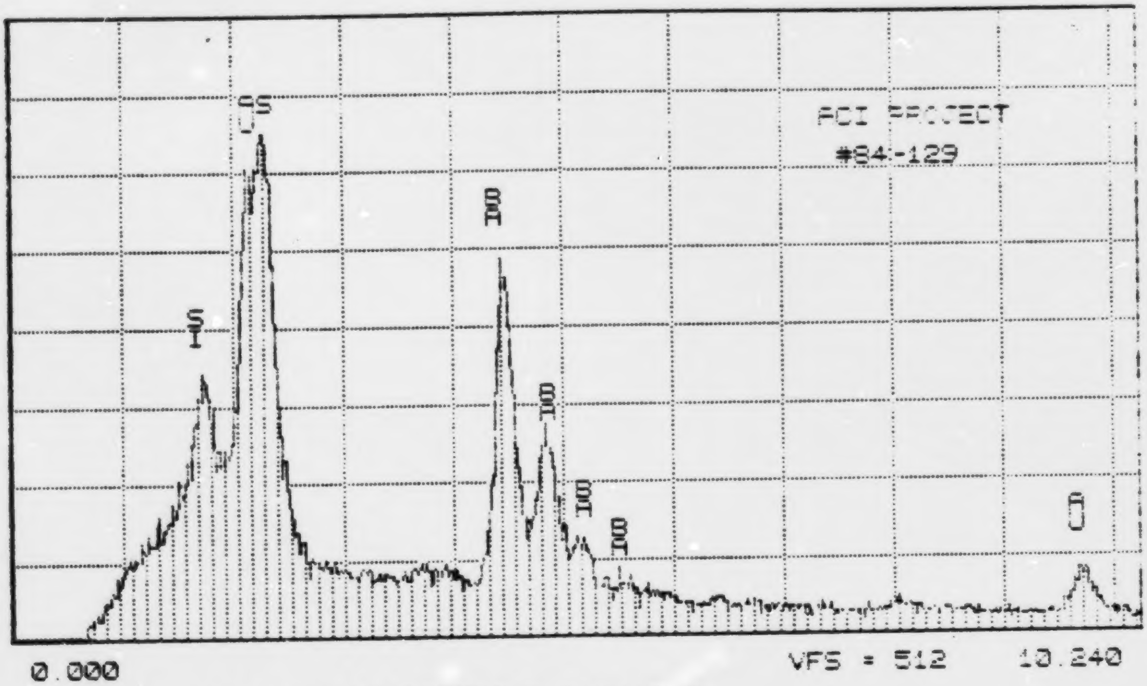


10   DUST 85-132 PARTICLE P 0410-01

19

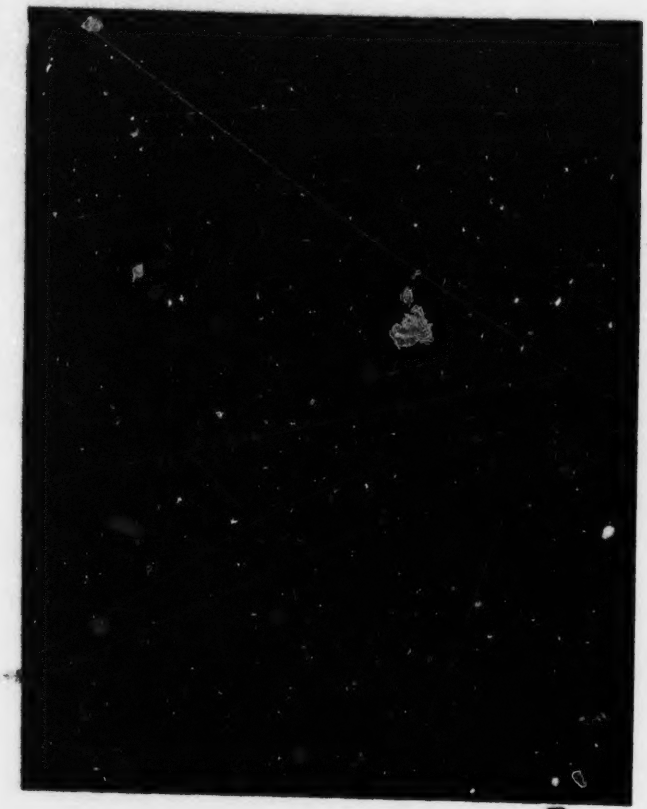
7a. PHOTOMICROGRAPHS: COAL DUST PARTICULATES ON MILLIPORE

1. Coal dust collected on Millipore filter #85-38 at the brusher work station. The filter has been carbon coated. x2000, BEI, 20kv. Numbers correspond to EDX spectra #1-14 for 10 second spot analyses.

2. Same filter: different field of view, corresponding to EDX #15-24.

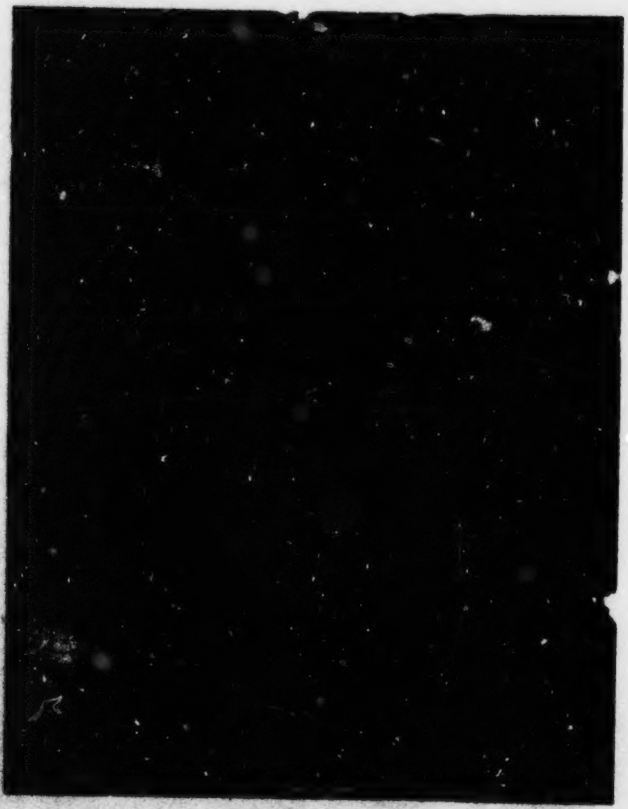
3. Coal dust collected on Millipore filter #85-53 at the coal wall face. Carbon coated, BEI, 20kv x 2000. Corresponding particles to EDX 25-33.

4. Same filter: different field of view corresponding to EDX #34-43.

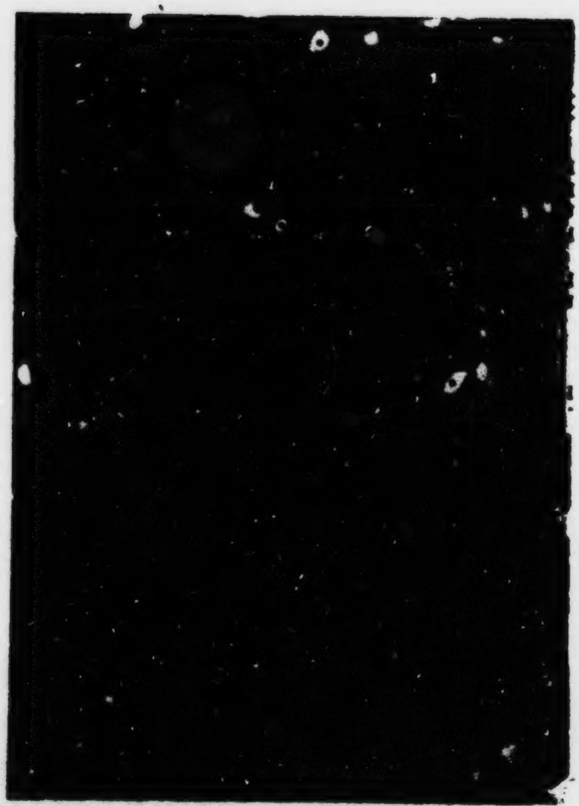


1

2



3



4

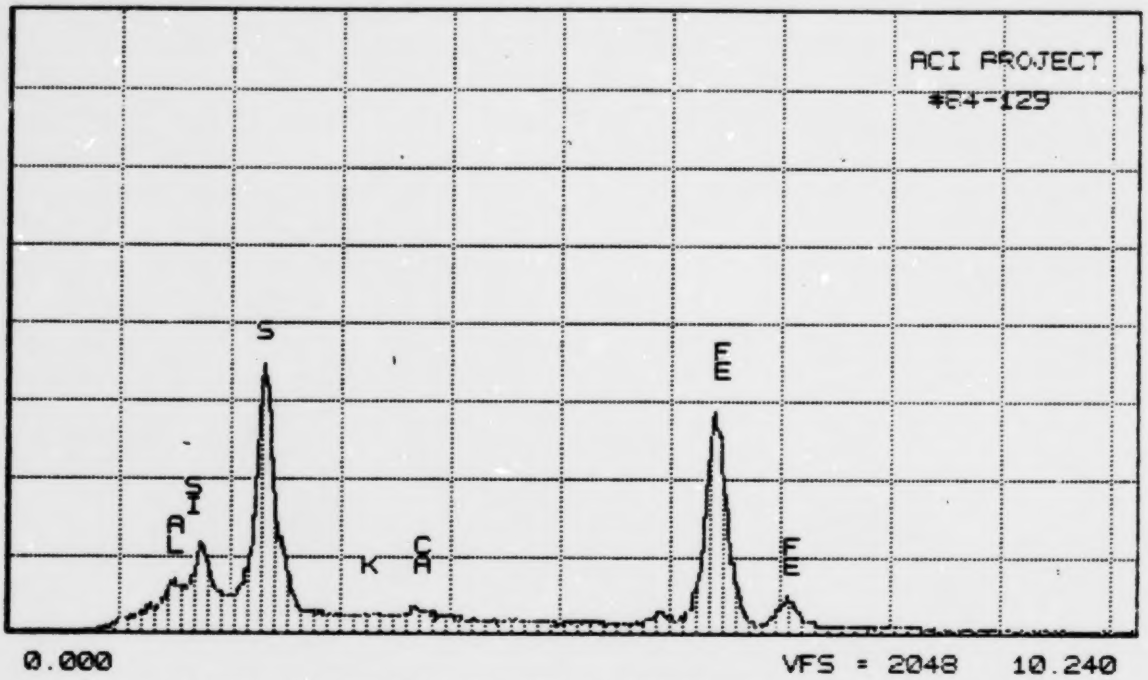
7b. EDX SPECTRA: COAL DUST PARTICULATES ON MILLIPORE

EDX spectra for particles on Millipore filter #85-38 taken as 10 second counts at 20kv. (photo #1):

Spectrum #	Particle #	Mineral I.D.
1	1	pyrite
2	2	illite
3	3	agglomerate of pyrite/illite
4	4	calcite
5	5	pyrite
6	6	coal maceral
7	7	illite
8	8	calcite
9	9	illite
10	10	calcite + illite
11	11	illite
12	12	calcite
13	13	pyrite
14	14	illite
Millipore filter #85-38, photo #2:		
15	1	pyrite
16	2	calcite
17	3	calcite
18	4	pyrite + trace calcite
19	5	illite
20	6	calcite
21	7	calcite
22	8	siderite (?)
23	9	calcite
24	10	coal maceral
Millipore filter #85-53, coal wall face respirable dust sample (photo #3):		
25	1	quartz or albite
26	2	chlorite (?)
27	3	calcite
28	4	illite (note Cl)
29	5	calcite
30	6	anhydrite(?) + illite (?)
31	7	coal + calcite
32	8	coal maceral
33	9	illite
Millipore filter #85-53, photo #4		
34	1	illite + halite
35	2	illite (note Ti)
36	3	kaolinite (?) + anhydrite (?)
37	4	chlorite (?)
38	5	illite
39	6	pyrite
40	7	quartz
41	8	calcite
42	9	chlorite (?)
43	10	quartz
44	Millipore filter backgroung	

ATLANTIC COAL INSTITUTE THU 07-MAR-85 16:57

Cursor: 0.000keV = 0



10 DUST #85-38 MIN ID 20 KV P0307-10 spot#1

1

ATLANTIC COAL INSTITUTE THU 07-MAR-85 16:59

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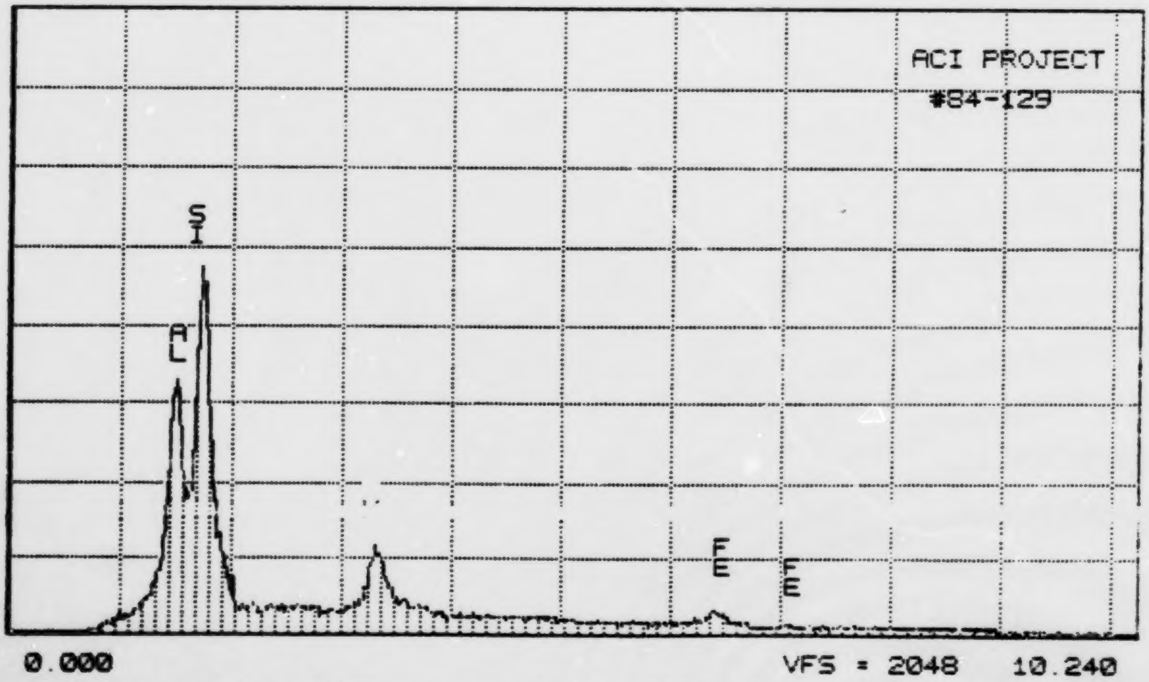

10 DUST #85-38 MIN ID 20 KV P0307-10 spot#2

2

ATLANTIC COAL INSTITUTE THU 07-MAR-85 17:01

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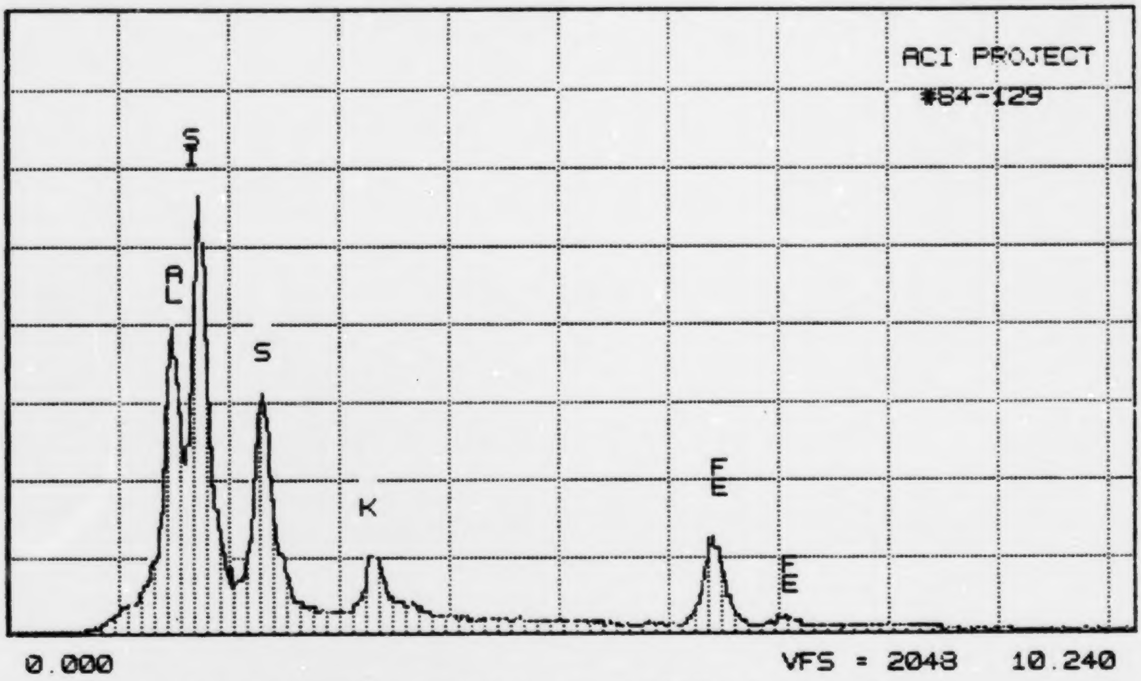

10 DUST #85-38 MIN ID 20 KV P0307-10 spot#3

3

ATLANTIC COAL INSTITUTE THU 07-MAR-85 17:03

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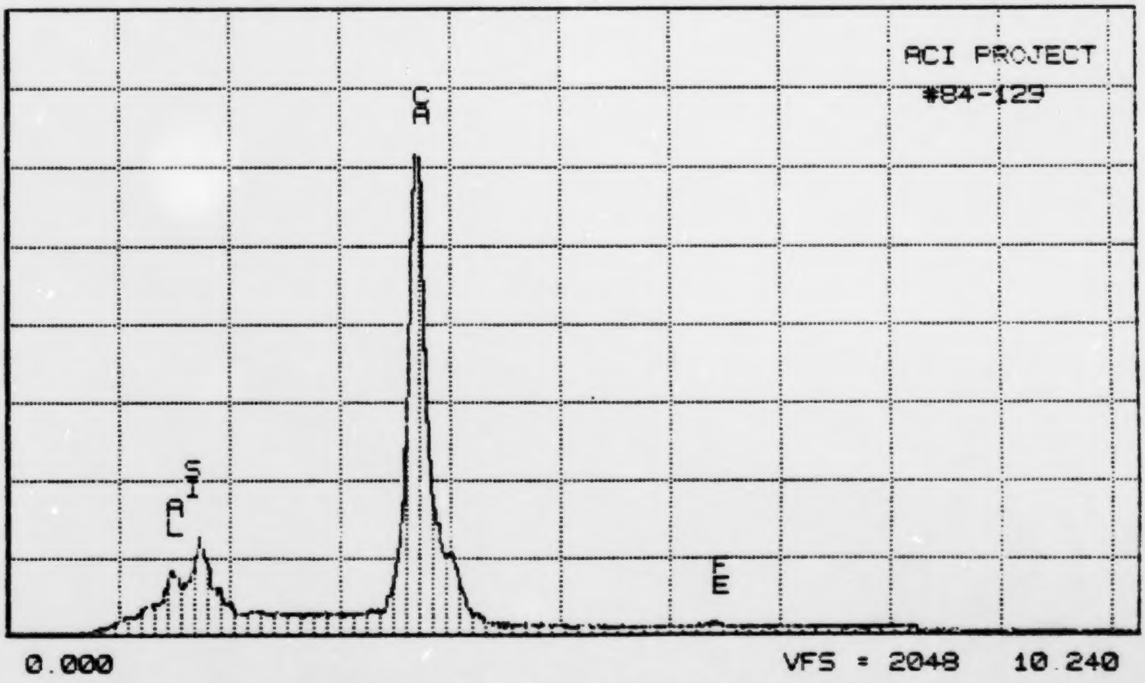

10 DUST #85-38 MIN ID 20 KV P0307-10 spot#4

4

ATLANTIC COAL INSTITUTE THU 07-MAR-85 17:05
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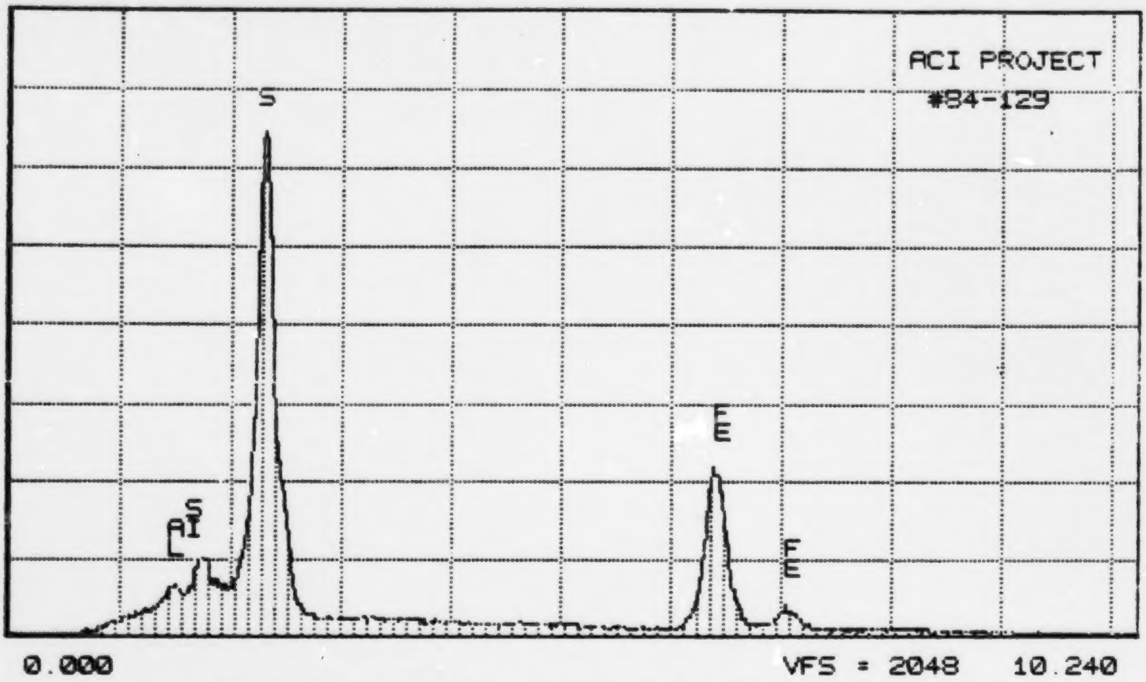

10 DUST #85-38 MIN ID 20 KV P0307-10 spot#5

5

ATLANTIC COAL INSTITUTE THU 07-MAR-85 17:07
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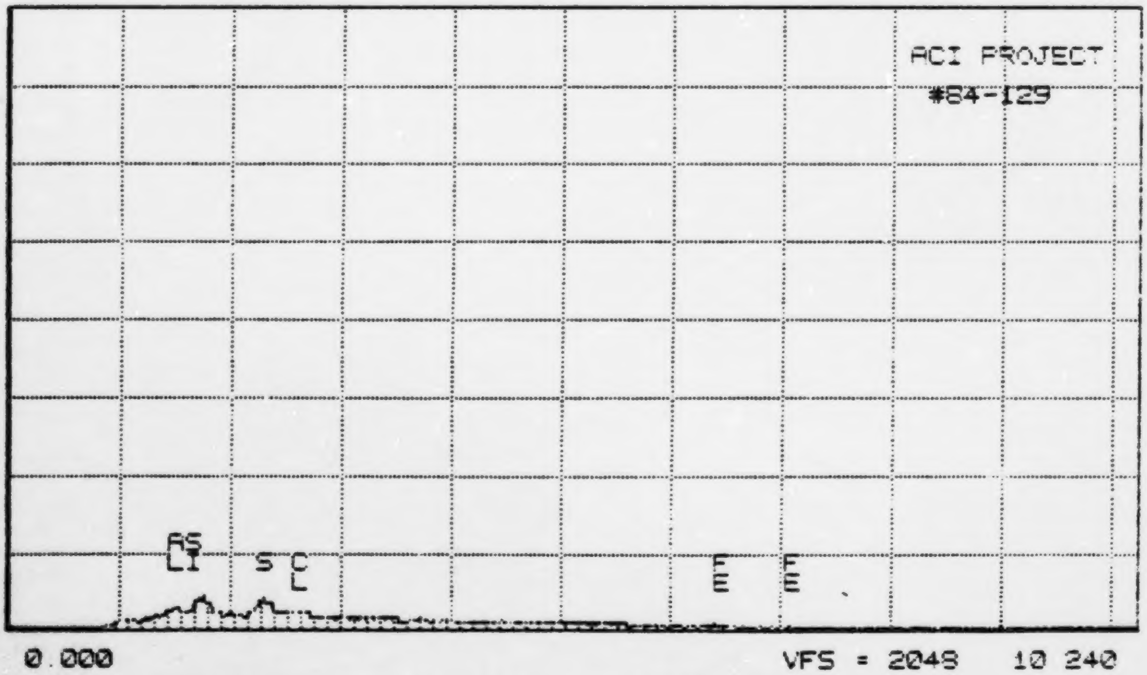

10 DUST #85-38 MIN ID 20 KV P0307-10 spot#6

6

ATLANTIC COAL INSTITUTE TUE 09-APR-85 20:19
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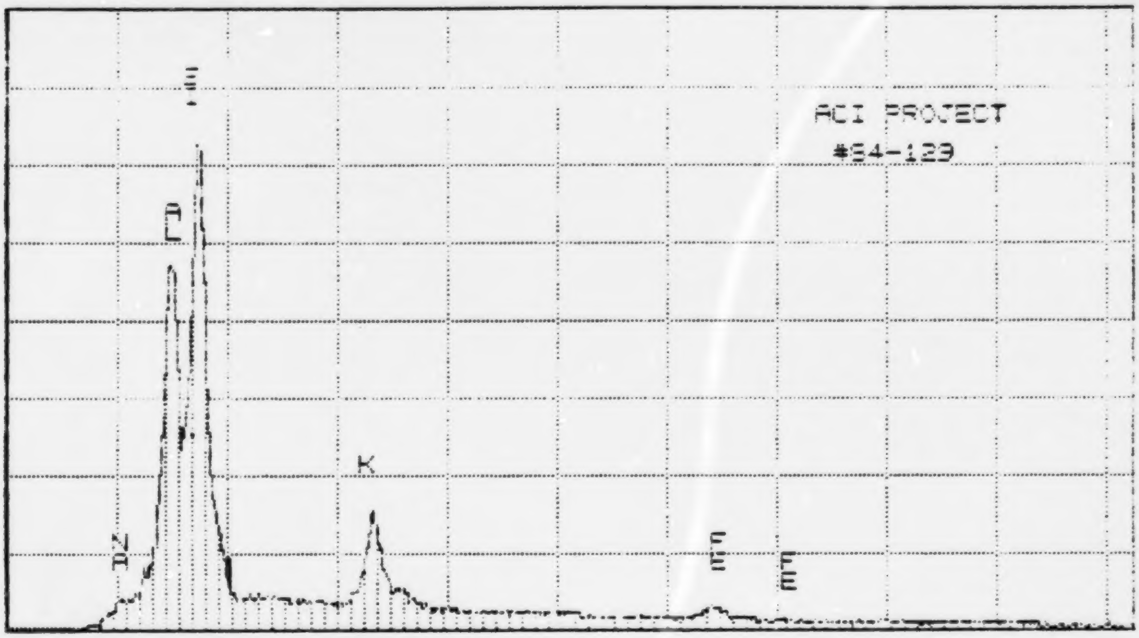

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10 DUST #85-38 MIN ID 20 KV P0307-10 spot#7

7

ATLANTIC COAL INSTITUTE THU 07-MAR-85 17:11
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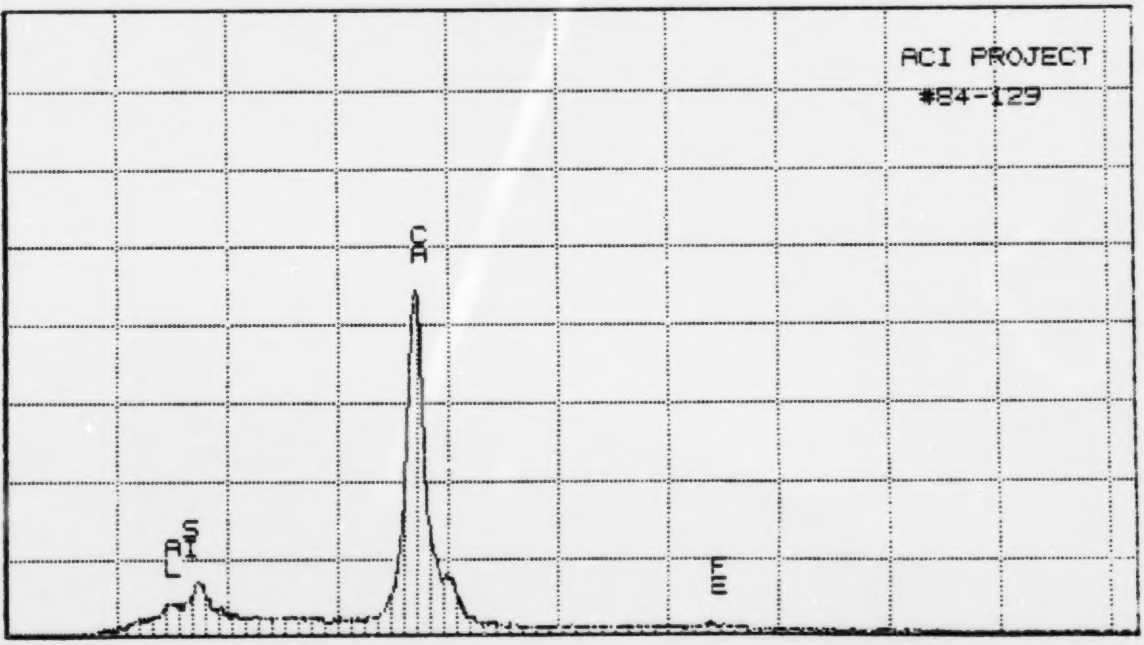

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10 DUST #85-38 MIN ID 20 KV P0307-10 spot#8

8

ATLANTIC COAL INSTITUTE THU 07-MAR-85 17:13

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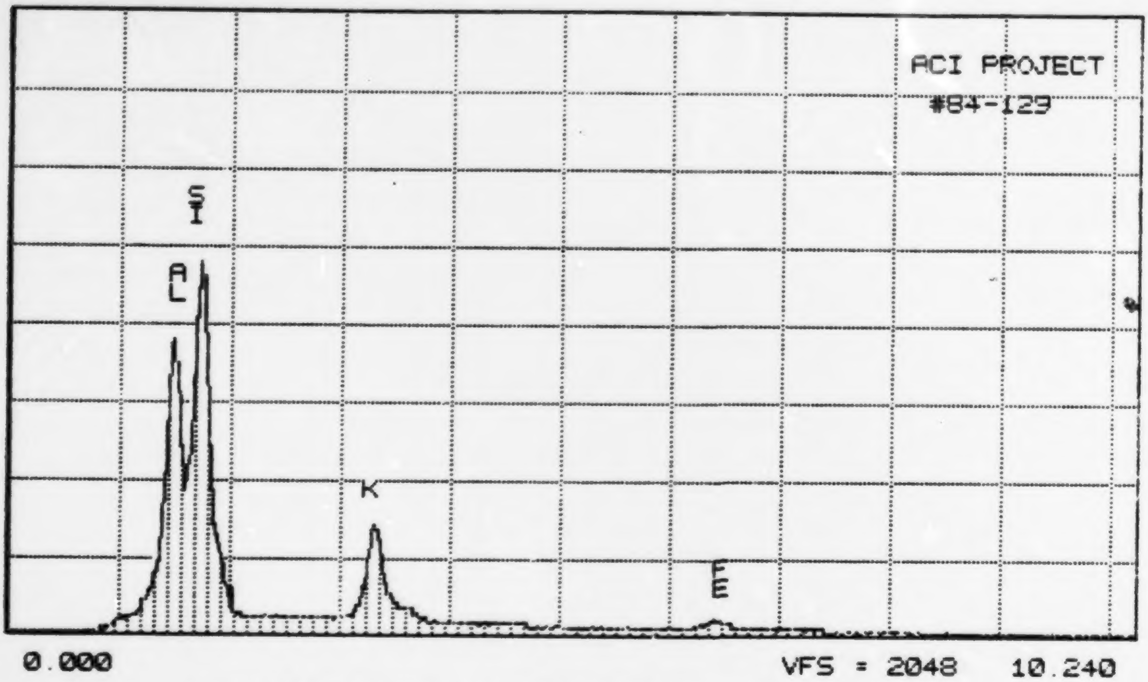

10 DUST #85-38 MIN ID 20 KV P0307-10 spot#9

9

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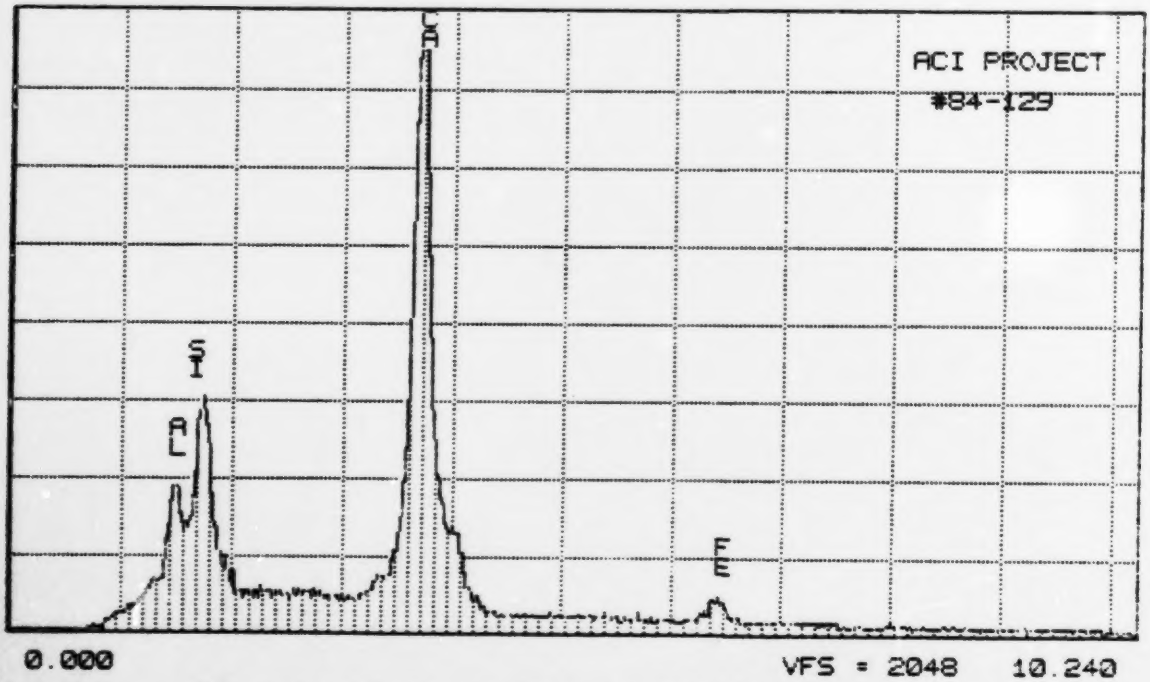

10 DUST #85-38 MIN ID 20 KV P0307-10 spot#10

10

ATLANTIC COAL INSTITUTE THU 07-MAR-85 17:17
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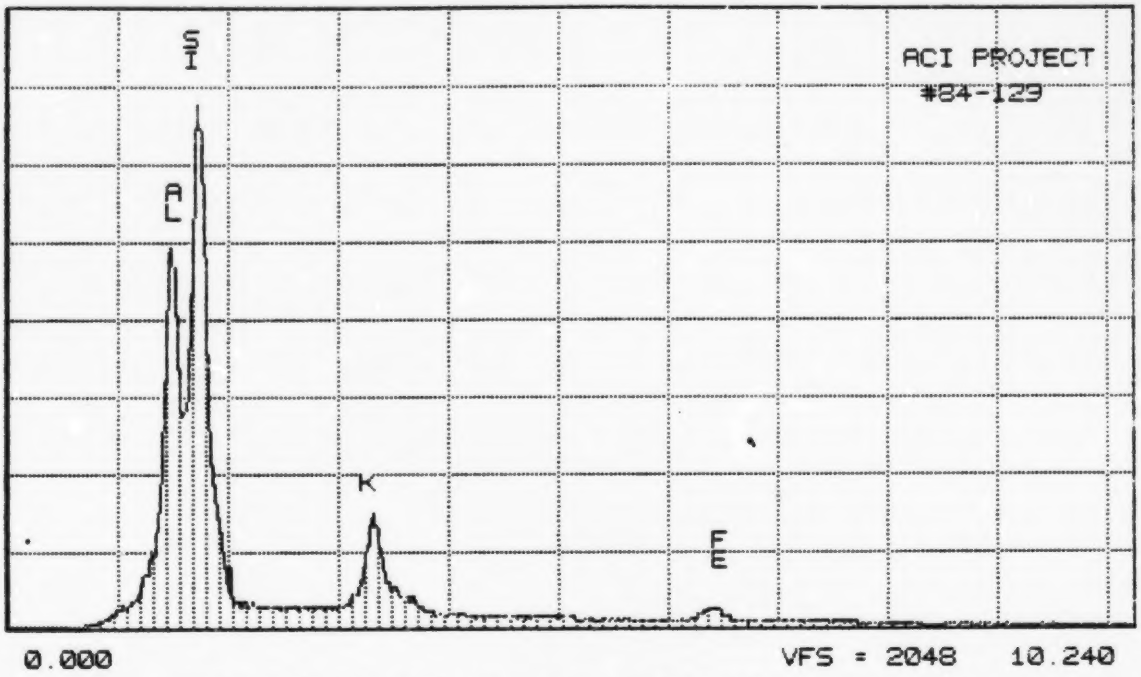

10 DUST #85-38 MIN ID 20 KV P0307-10 spot#11

11

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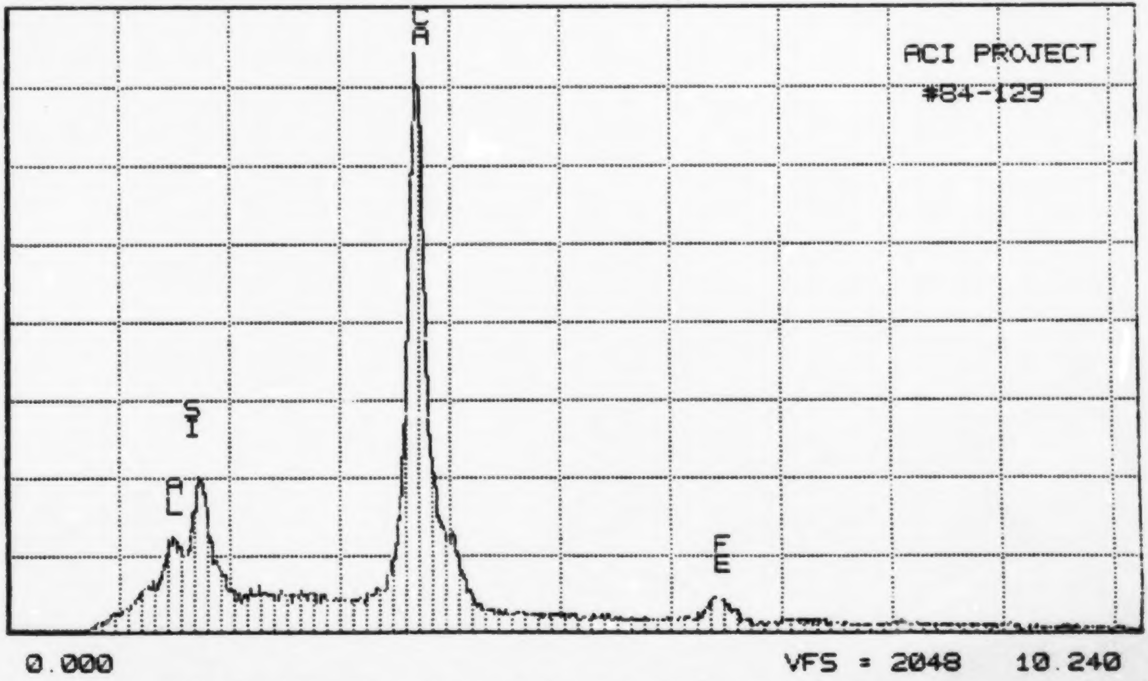

10 DUST #85-38 MIN ID 20 KV P0307-10 spot#12

12

ATLANTIC COAL INSTITUTE THU 07-MAR-85 17:22
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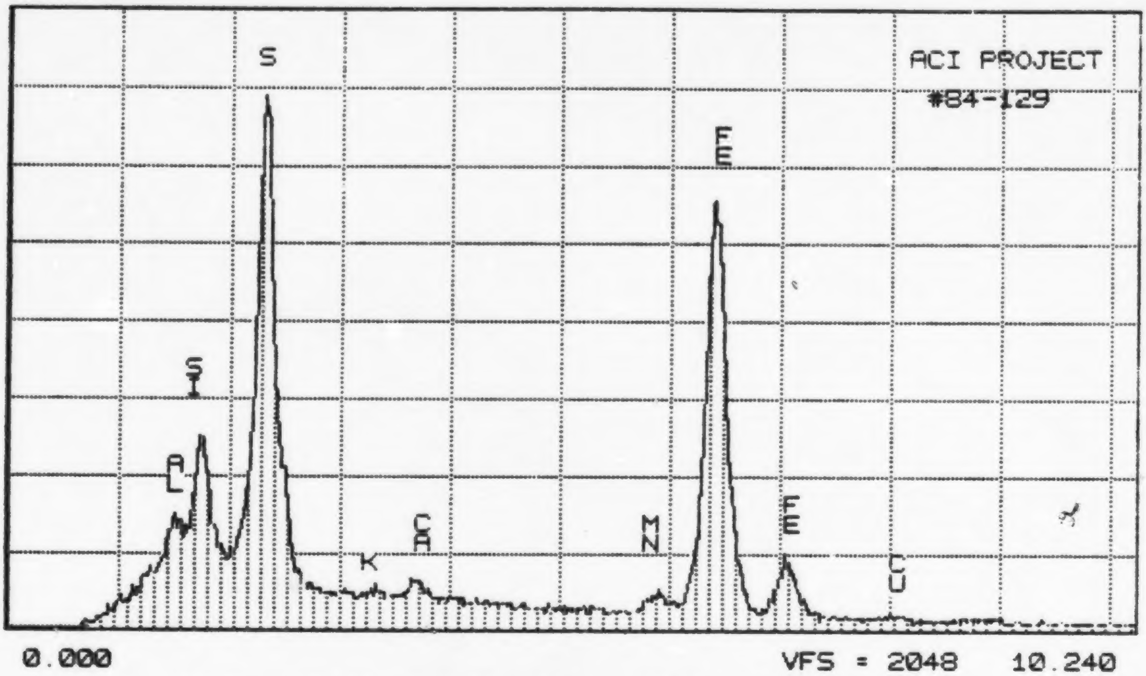

10 DUST #85-38 MIN ID 20 KV P0307-10 spot#13

13

ATLANTIC COAL INSTITUTE THU 07-MAR-85 17:24
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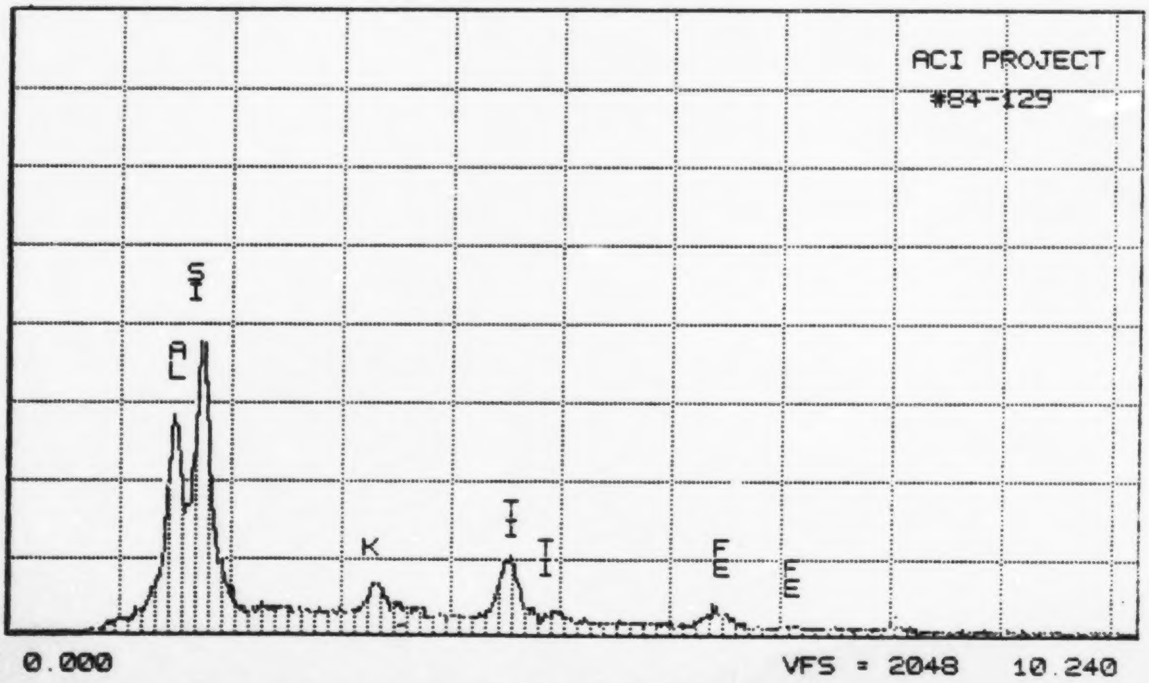

10 DUST #85-38 MIN ID 20 KV P0307-10 spot#14

14

ATLANTIC COAL INSTITUTE THU 07-MAR-85 17:28

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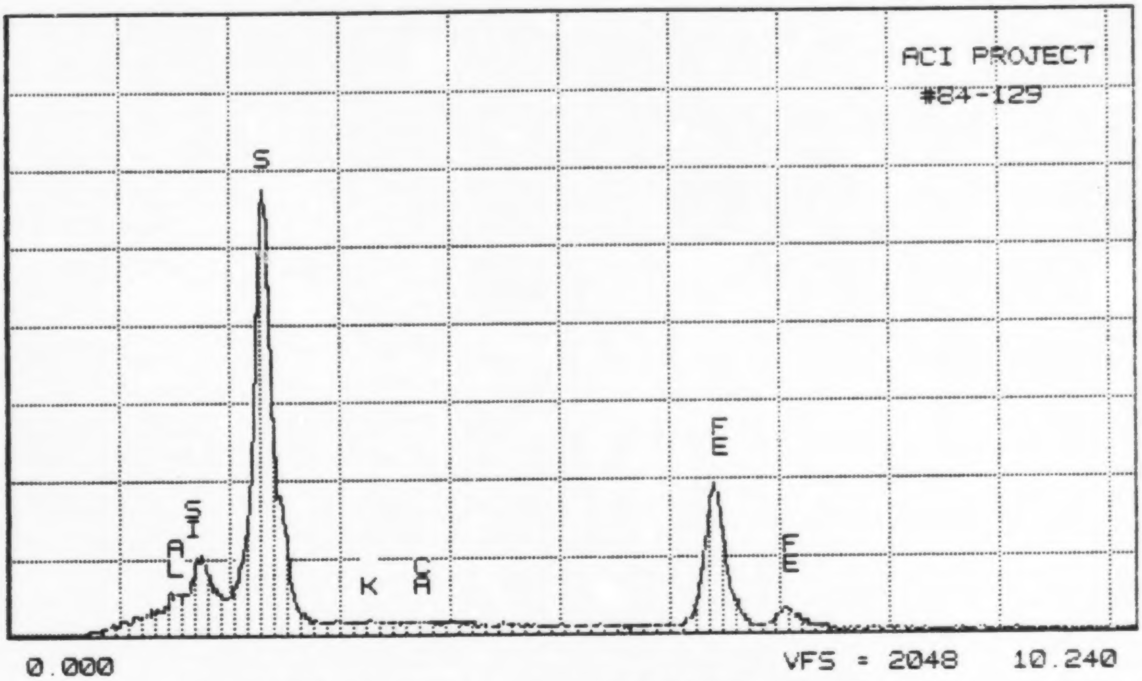

10 DUST #85-38 MIN ID 20kV P0307-15 spot#1

15

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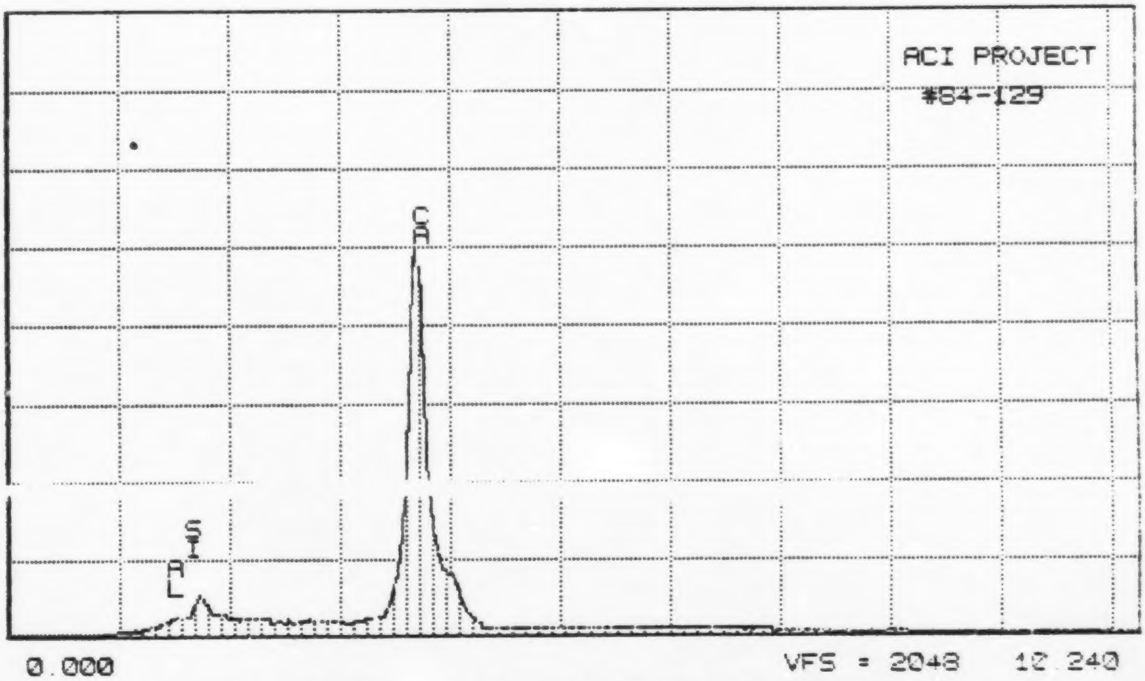

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16

ATLANTIC COAL INSTITUTE THU 07-MAR-85 17:32
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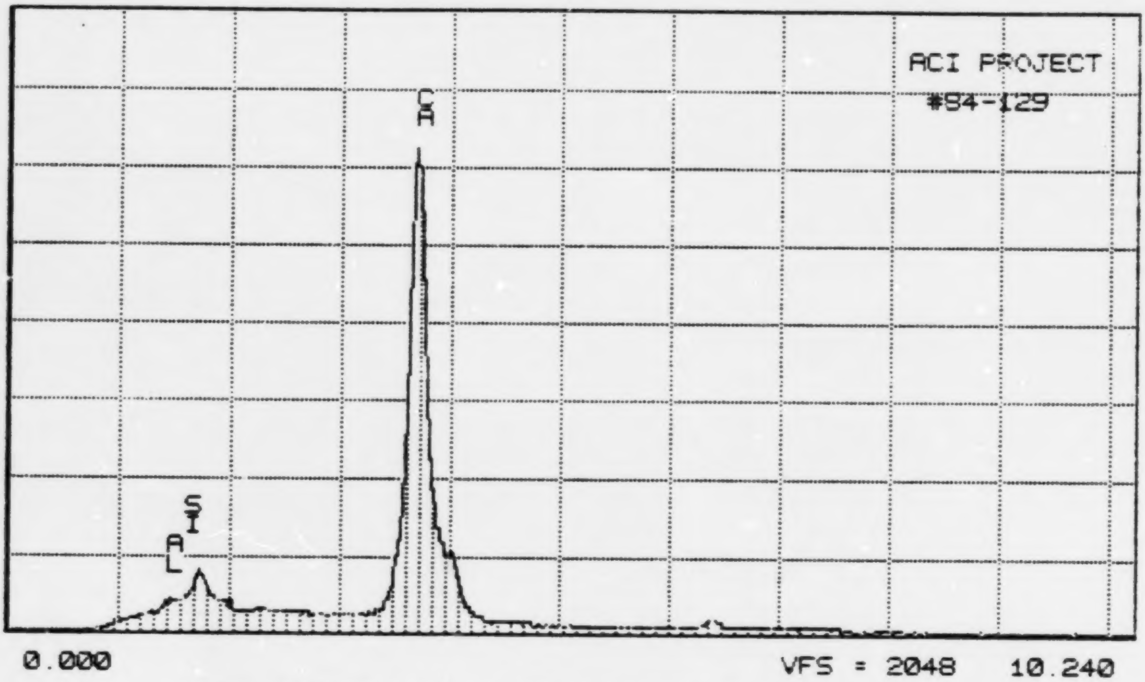

17

10 DUST #85-38 MIN ID 20kV P0307-15 spot#3

ATLANTIC COAL INSTITUTE THU 07-MAR-85 17:34
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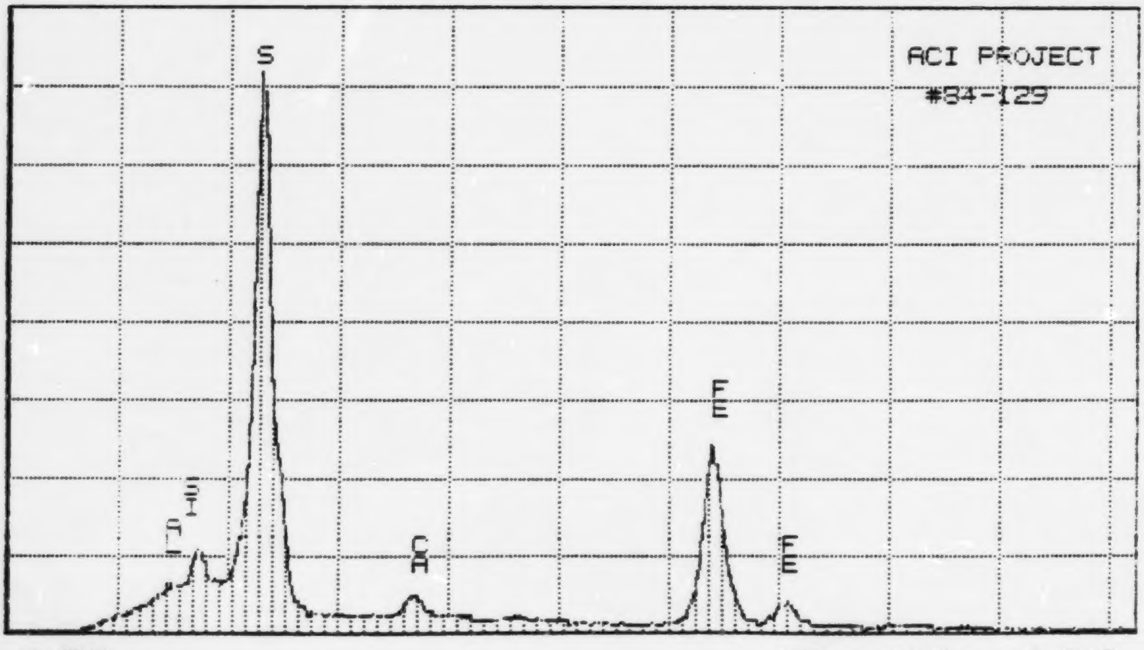

18

10 DUST #85-38 MIN ID 20kV P0307-15 spot#4

ATLANTIC COAL INSTITUTE THU 07-MAR-85 17:36

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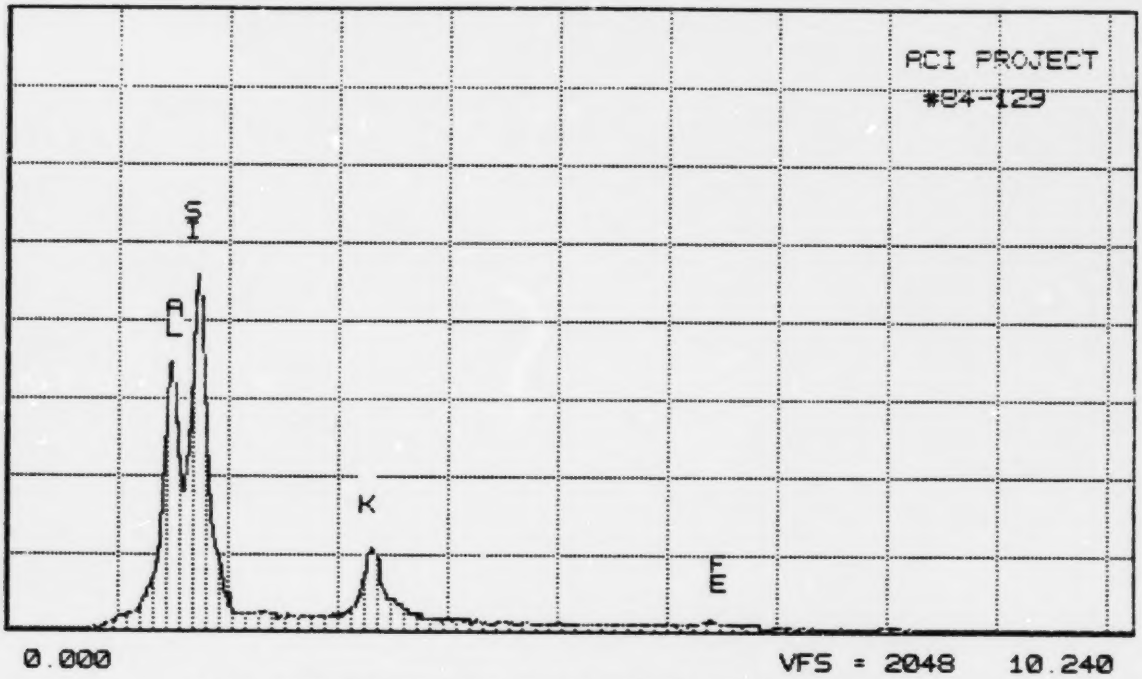

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19

ATLANTIC COAL INSTITUTE THU 07-MAR-85 17:38

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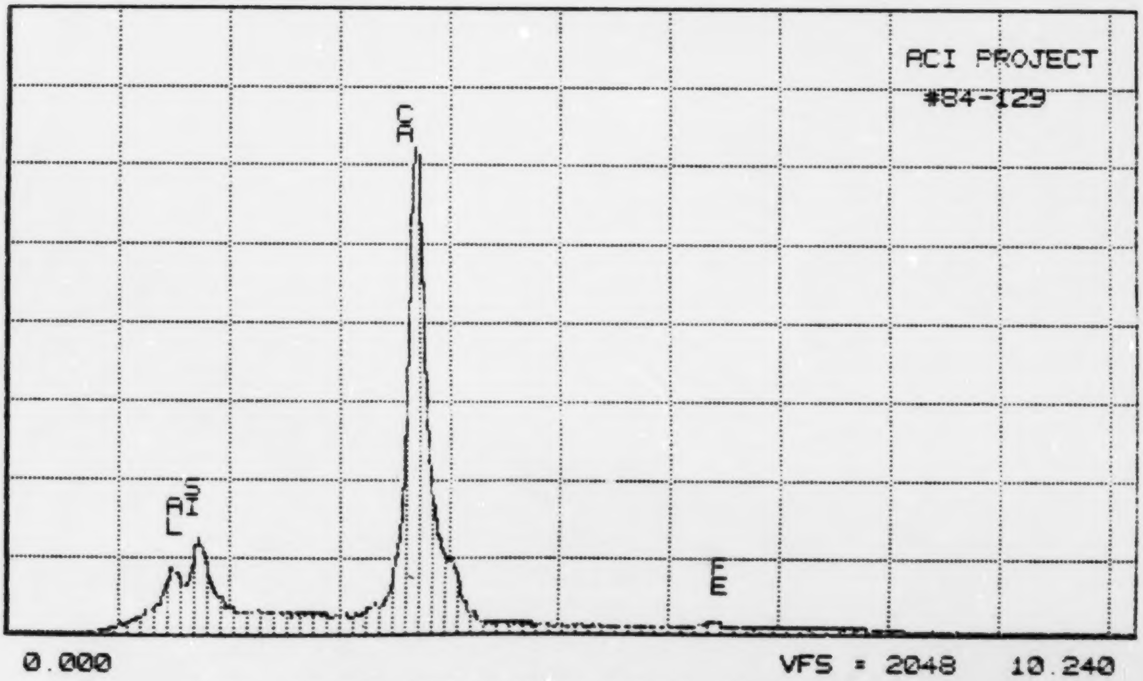

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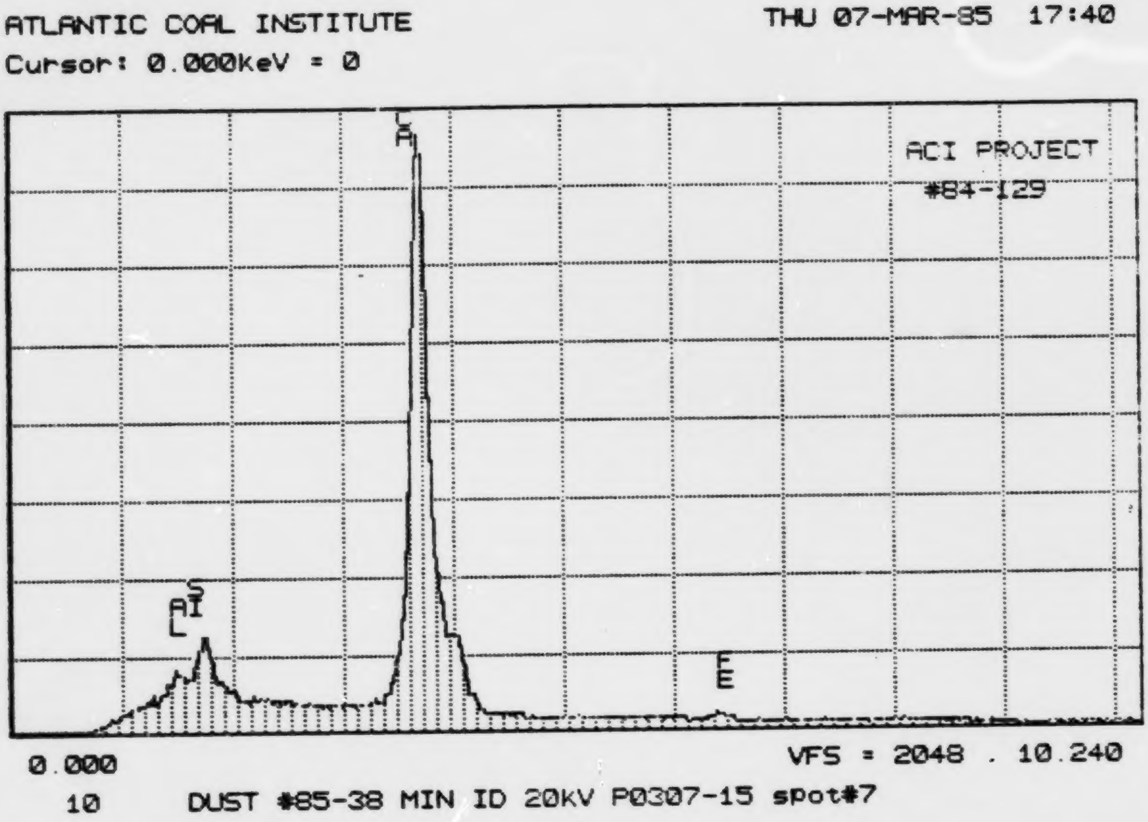
20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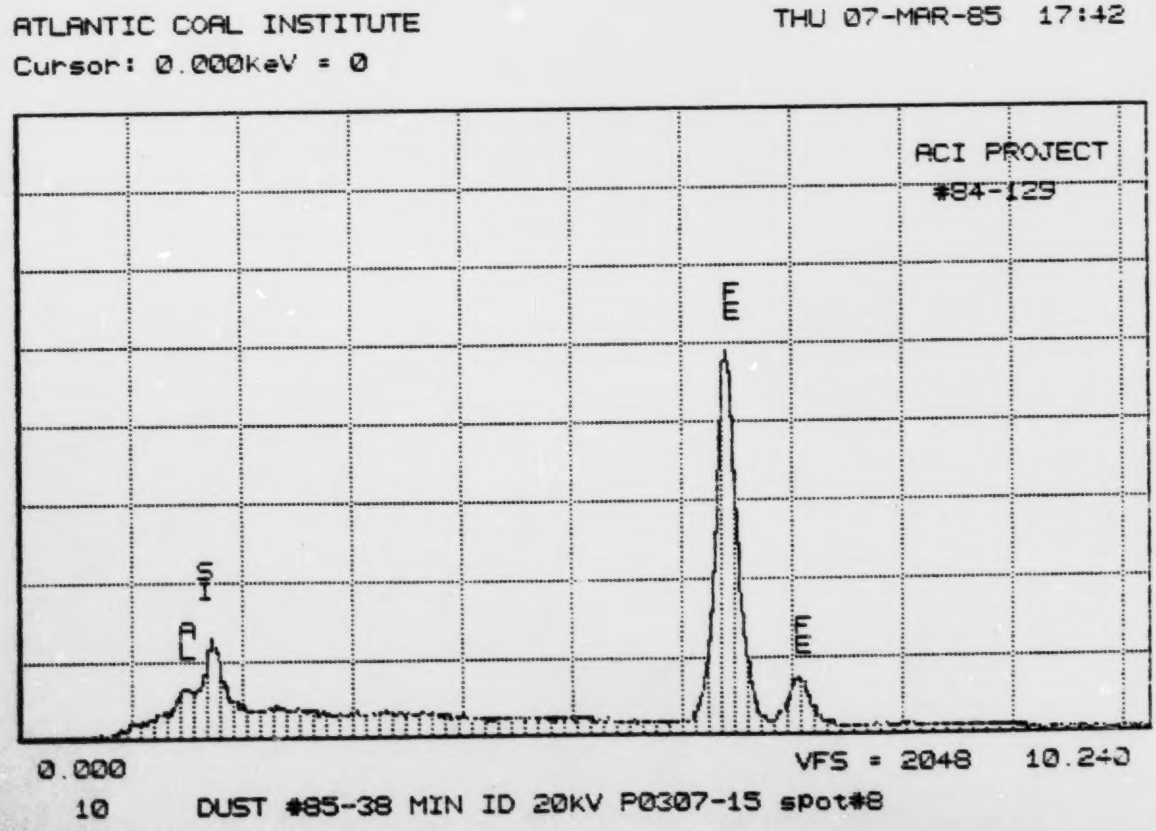
ACI PROJECT #84-129

0.000 VFS = 2048 10.240

10 DUST #85-38 MIN ID 20kV P0307-15 spot#7

21


ATLANTIC COAL INSTITUTE THU 07-MAR-85 17:42

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ACI PROJECT #84-129

0.000 VFS = 2048 10.240

10 DUST #85-38 MIN ID 20kV P0307-15 spot#8

22

ATLANTIC COAL INSTITUTE THU 07-MAR-85 17:44
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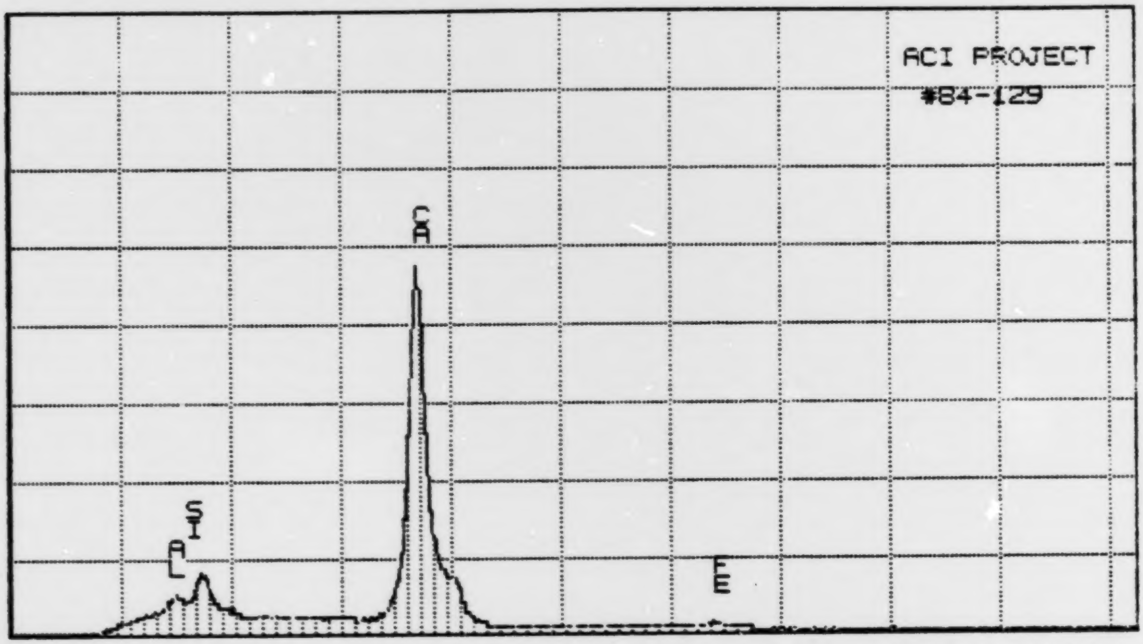

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23

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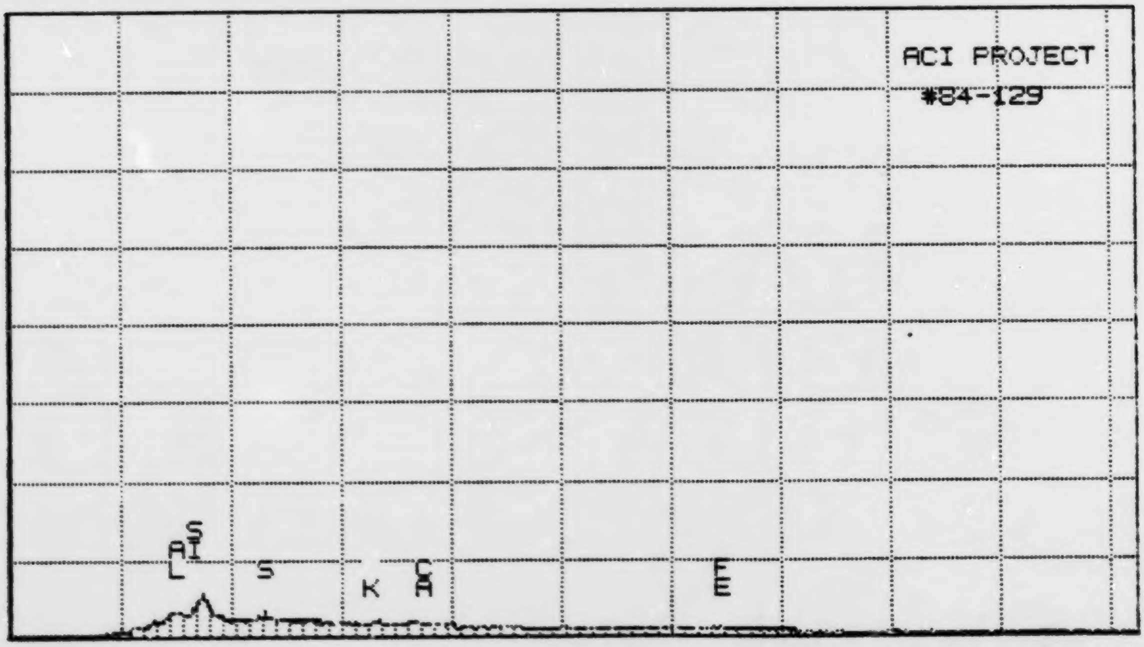

0.000 VFS = 2048 10.240
10 DUST #85-38 MIN ID 20kV P0307-15 spot#10

24

ATLANTIC COAL INSTITUTE TUE 12-MAR-85 14:11
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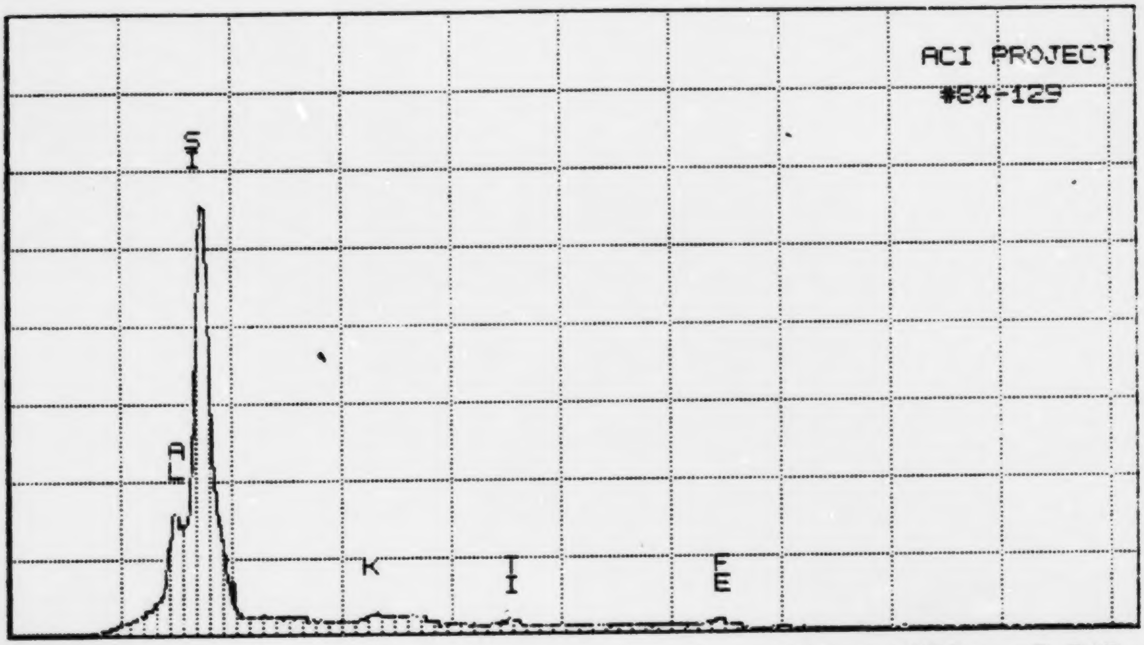

0.000 VFS = 4096 10.240
10 DUST #85-53 MIN ID 20kV P#0308-12 spot#1

25

ATLANTIC COAL INSTITUTE TUE 12-MAR-85 14:13
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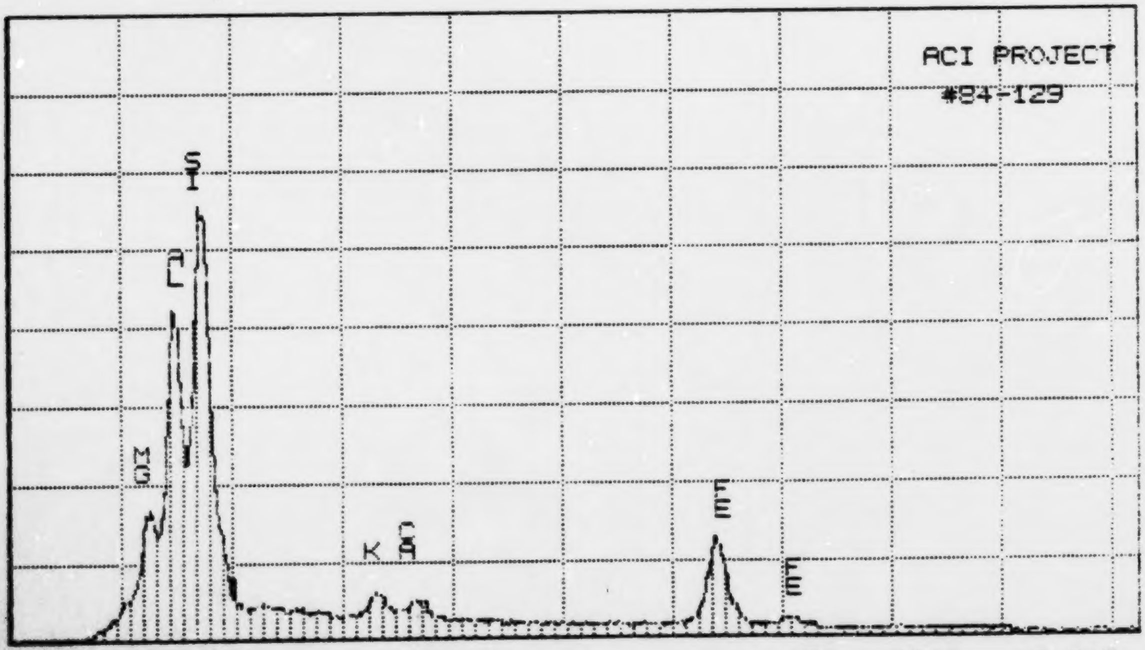
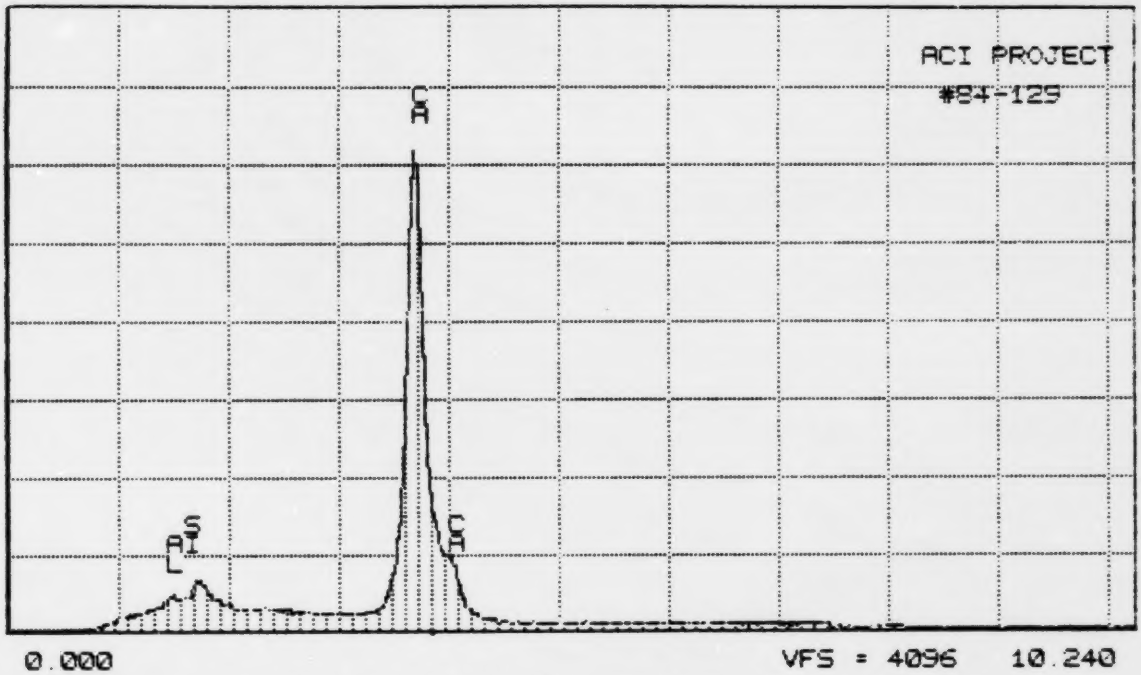


0.000 VFS = 2048 10.240
10 DUST #85-53 MIN ID 20kV P#0308-12 spot#2

26

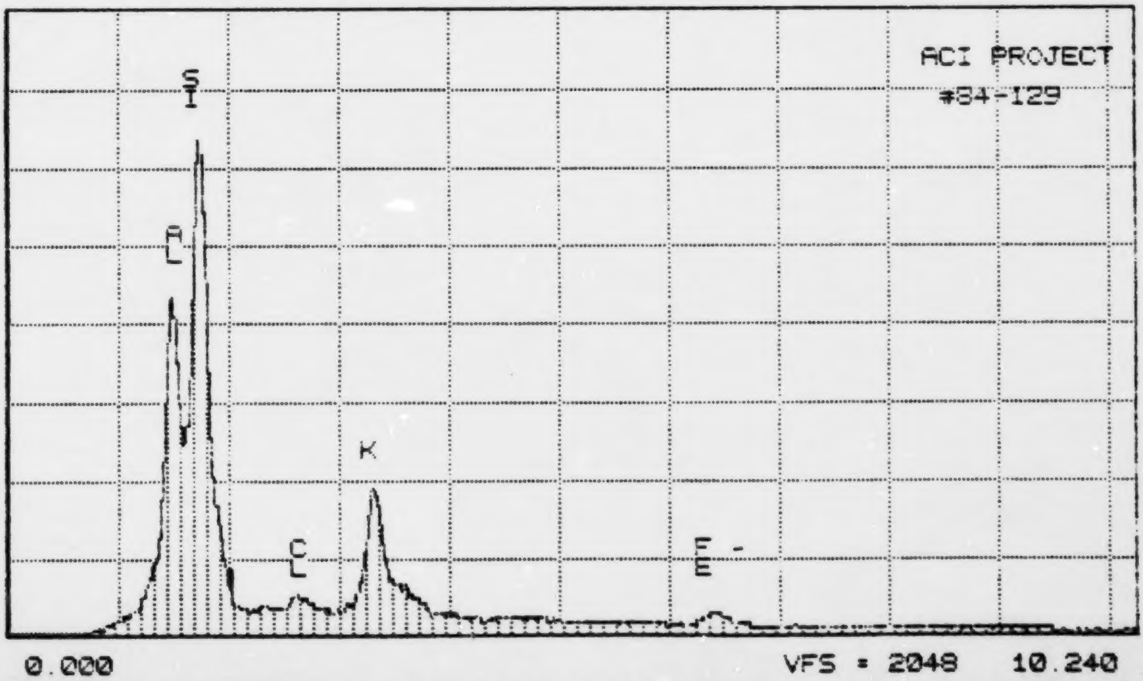
ATLANTIC COAL INSTITUTE TUE 12-MAR-85 16:28
Cursor: 0.000keV = 0



10 DUST #85-53 MIN ID 20KV P#0308-12 spot#3

27

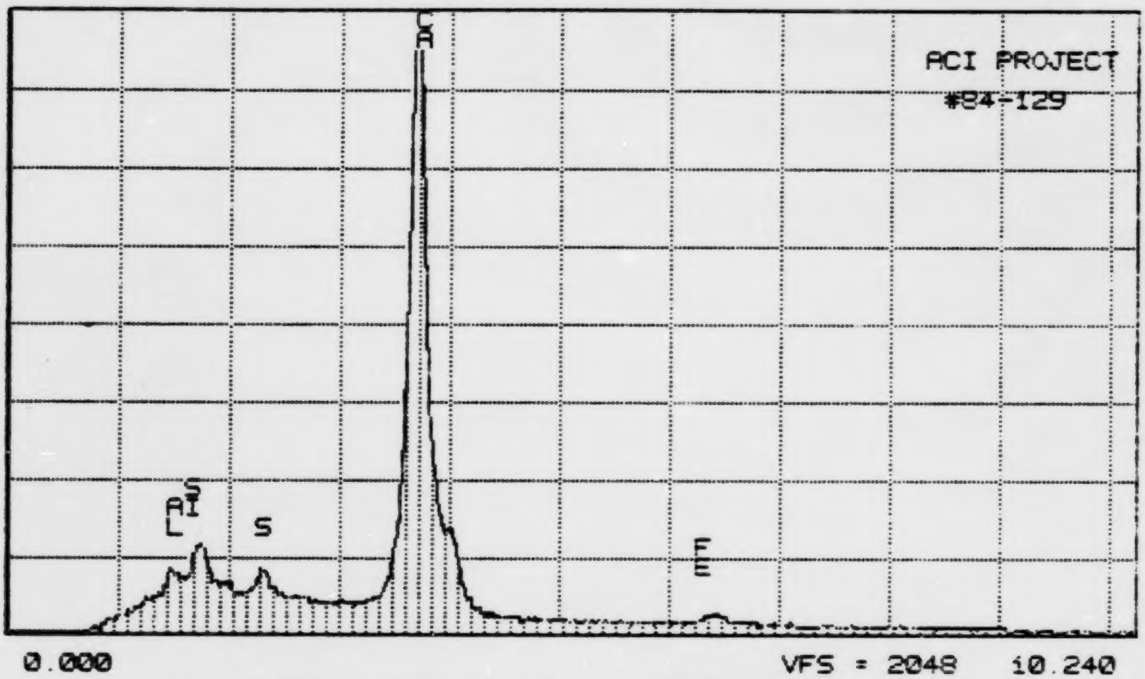
ATLANTIC COAL INSTITUTE TUE 12-MAR-85 16:30
Cursor: 0.000keV = 0



10 DUST #85-53 MIN ID 20KV P#0308-12 spot#4

28

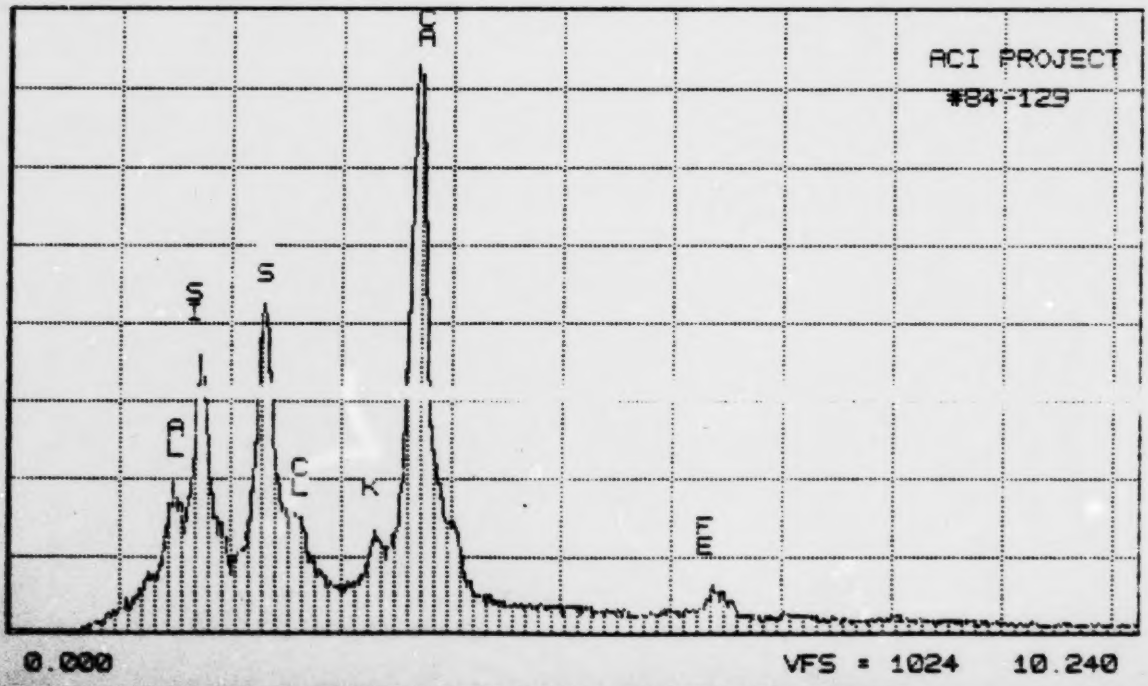
ATLANTIC COAL INSTITUTE TUE 12-MAR-85 16:33
Cursor: 0.000keV = 0



10 DUST #85-53 MIN ID 20KV P#0308-12 spot#5

29

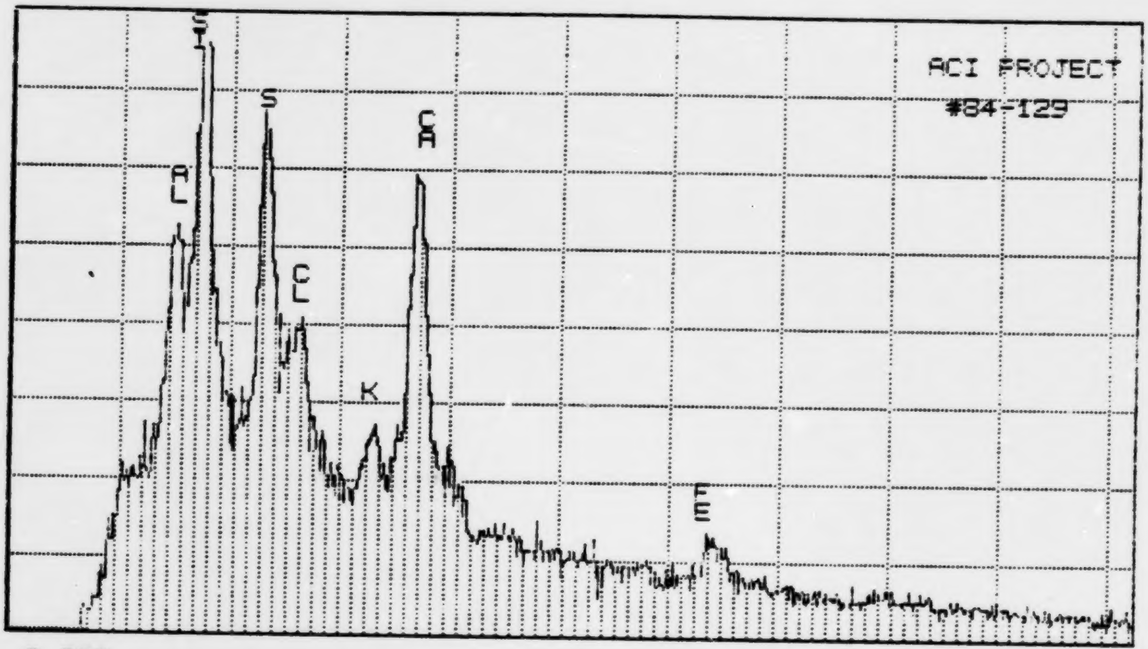
ATLANTIC COAL INSTITUTE TUE 12-MAR-85 16:35
Cursor: 0.000keV = 0



10 DUST #85-53 MIN ID 20KV P#0308-12 spot#6.

30

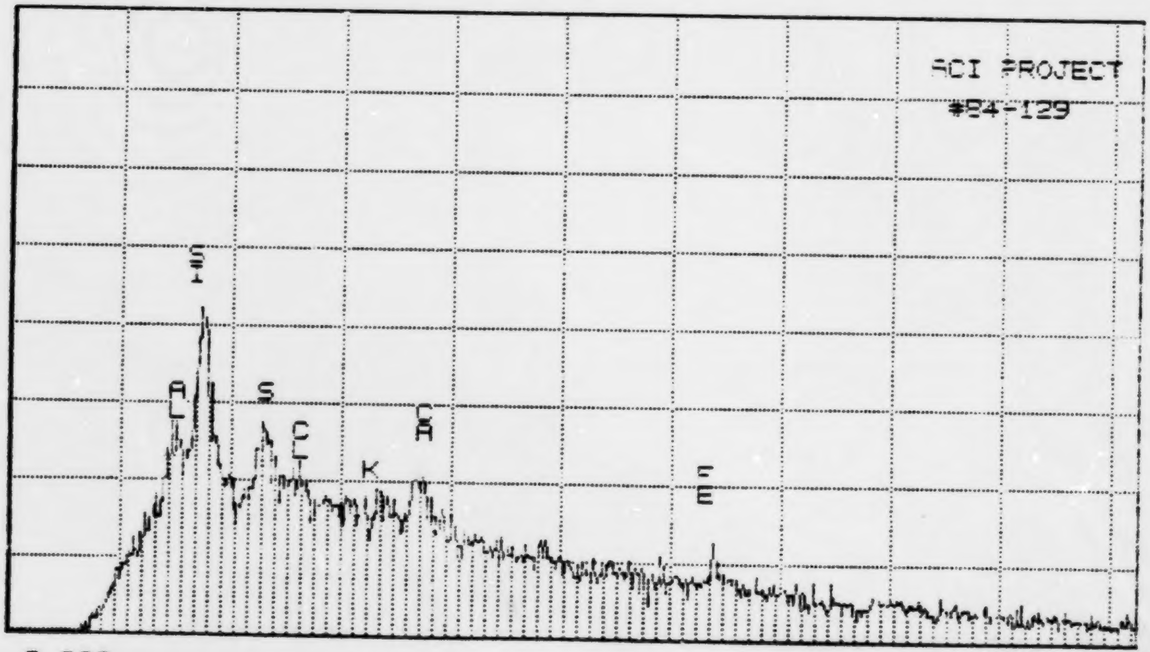
ATLANTIC COAL INSTITUTE TUE 12-MAR-85 16:37
Cursor: 0.000keV = 0



0.000 VFS = 256 10.240
10 DUST #85-53 MIN ID 20kV P#0308-12 spot#7

31

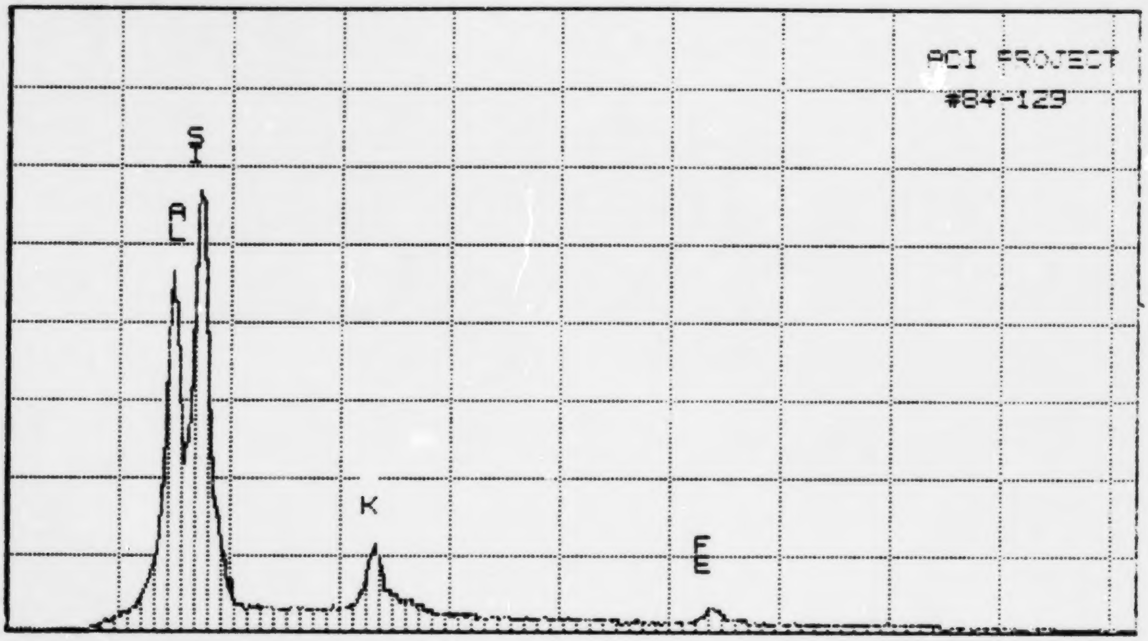
ATLANTIC COAL INSTITUTE TUE 12-MAR-85 16:29
Cursor: 0.000keV = 0



0.000 VFS = 256 10.240
10 DUST #85-53 MIN ID 20kV P#0308-12 spot#8

32

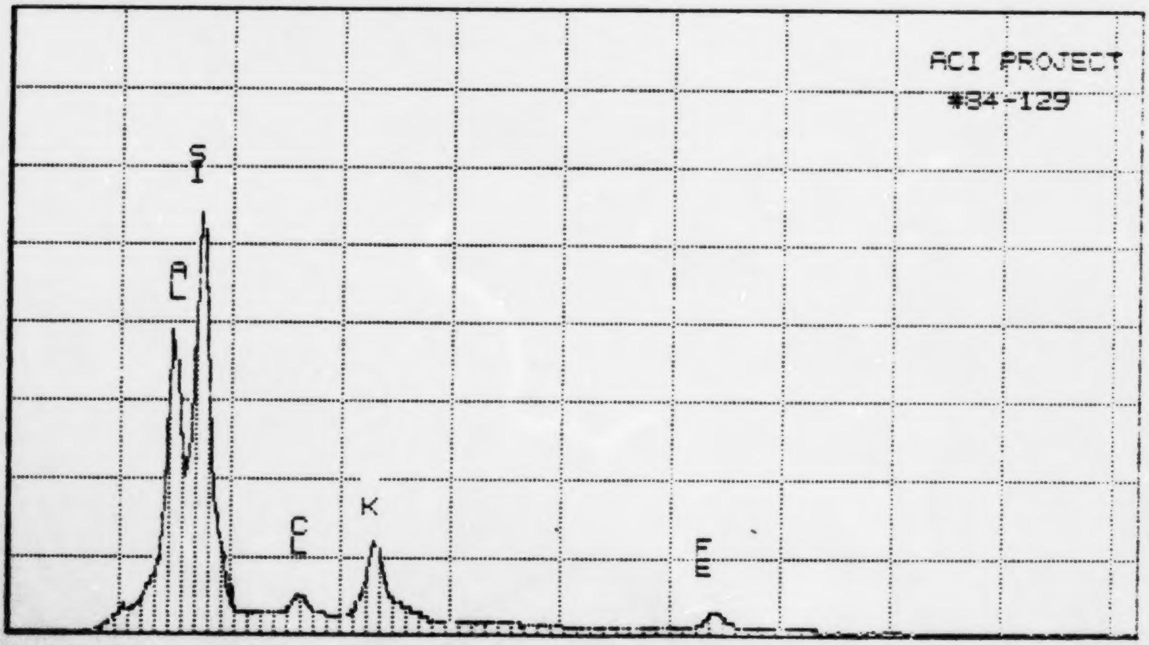
ATLANTIC COAL INSTITUTE TUE 12-MAR-85 16:41
Cursor: 0.000keV = 0



0.000 VFS = 2048 10.240
10 DUST #85-53 MIN ID 20kV P#0308-12 spot#9

33

ATLANTIC COAL INSTITUTE TUE 12-MAR-85 16:51
Cursor: 0.000keV = 0

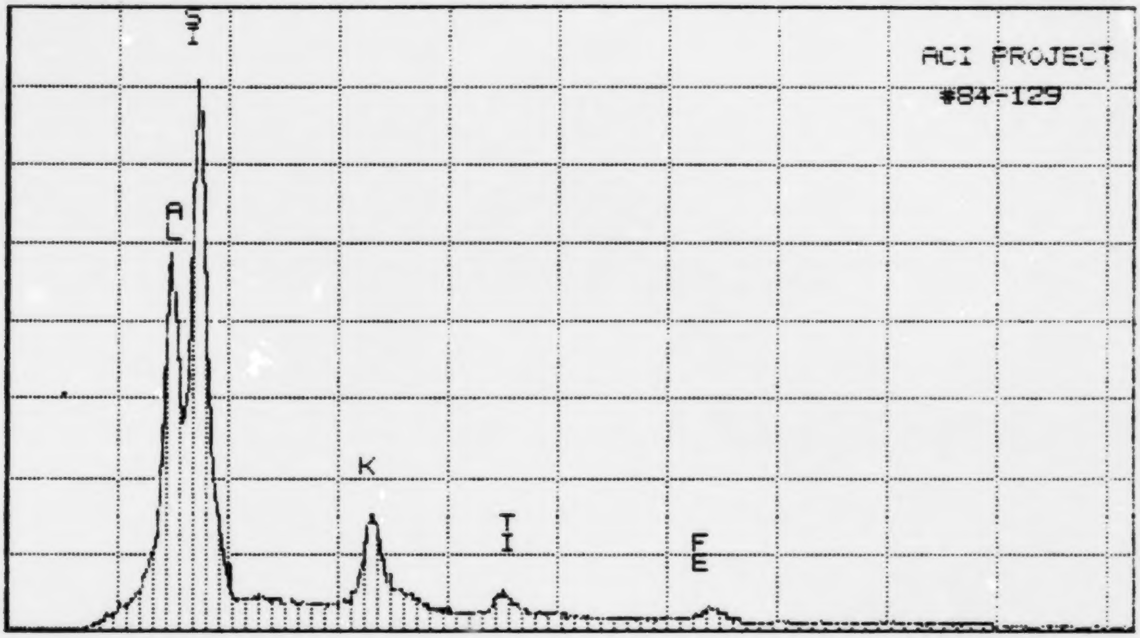


0.000 VFS = 4096 10.240
10 DUST #85-53 20KV MIN ID P#0308-14 spot #1

34

ATLANTIC COAL INSTITUTE TUE 12-MAR-85 16:54

Cursor: 0.000keV = 0



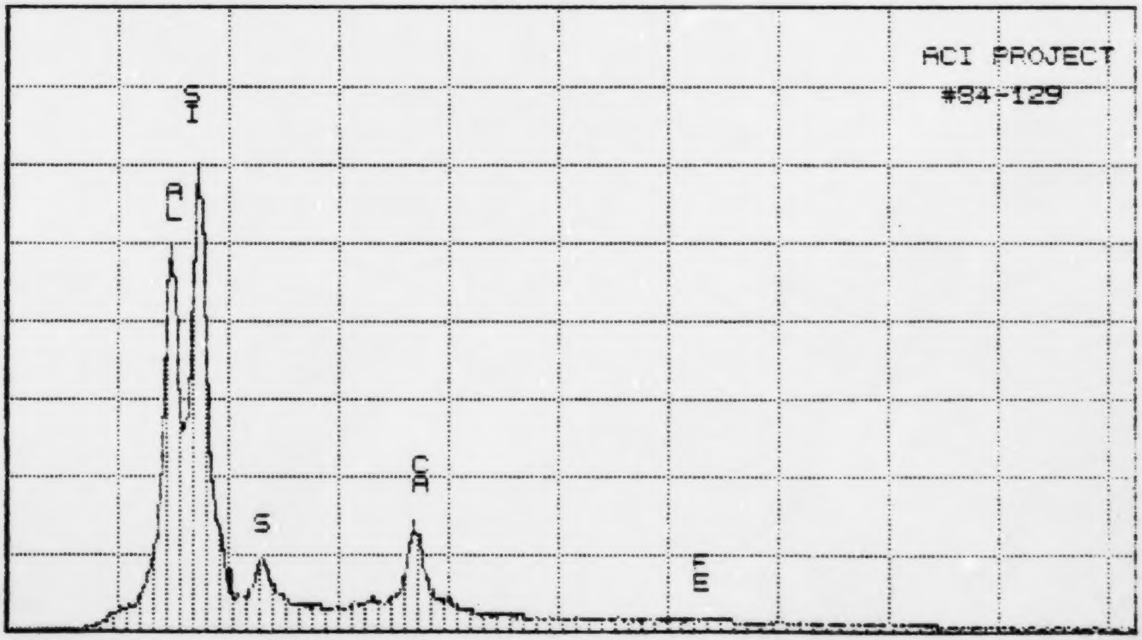
0.000 VFS = 2048 10.240

10 DUST #85-53 20kV MIN ID P#0308-14 spot #2

35

ATLANTIC COAL INSTITUTE TUE 12-MAR-85 16:56

Cursor: 0.000keV = 0



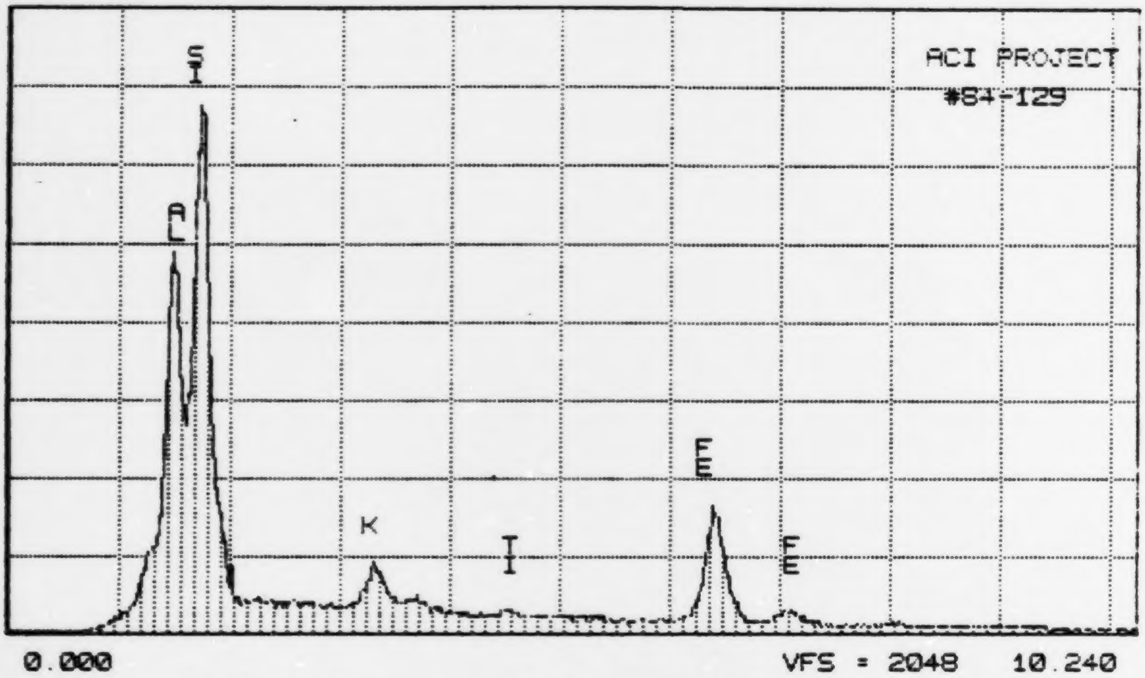
0.000 VFS = 2048 10.240

10 DUST #85-53 20kV MIN ID P#0308-14 spot #3

36

ATLANTIC COAL INSTITUTE TUE 12-MAR-85 16:58

Cursor: 0.000keV = 0

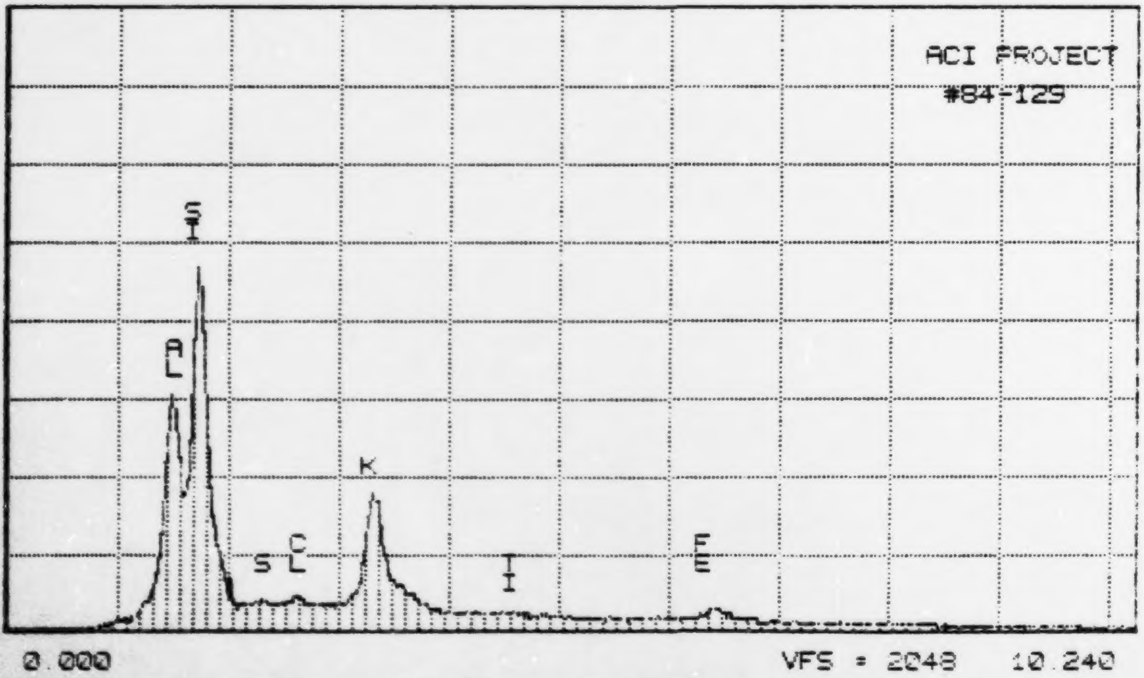


10 DUST #85-53 20KV MIN ID P#0308-14 spot #4

37

ATLANTIC COAL INSTITUTE TUE 12-MAR-85 17:00

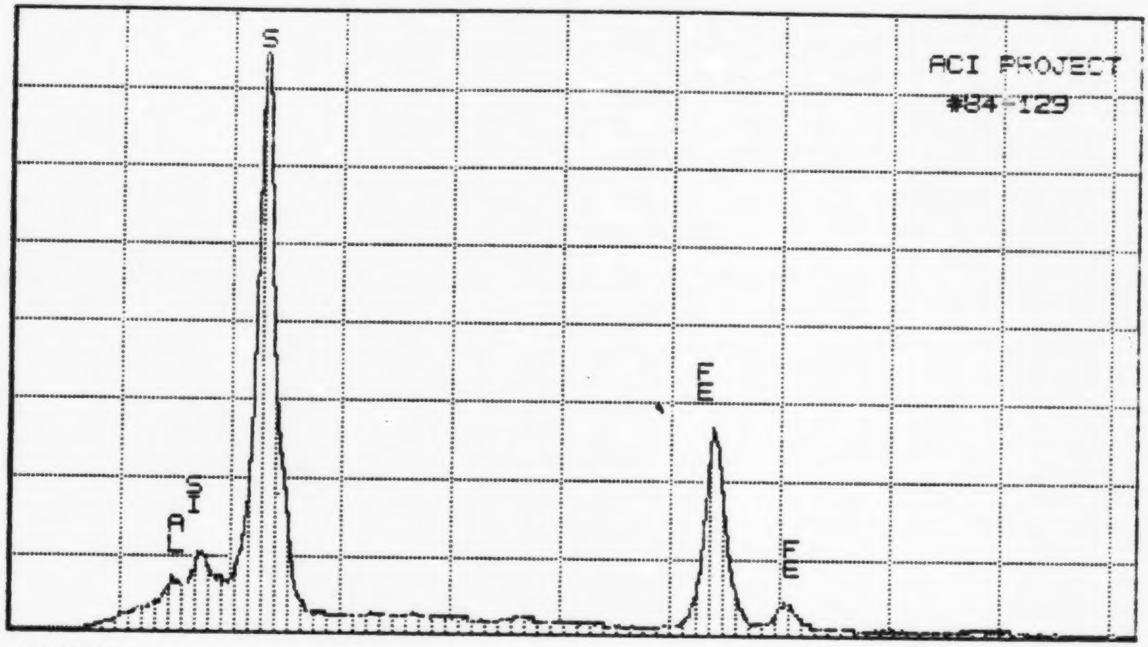
Cursor: 0.000keV = 0



10 DUST #85-53 20KV MIN ID P#0308-14 spot #5

38

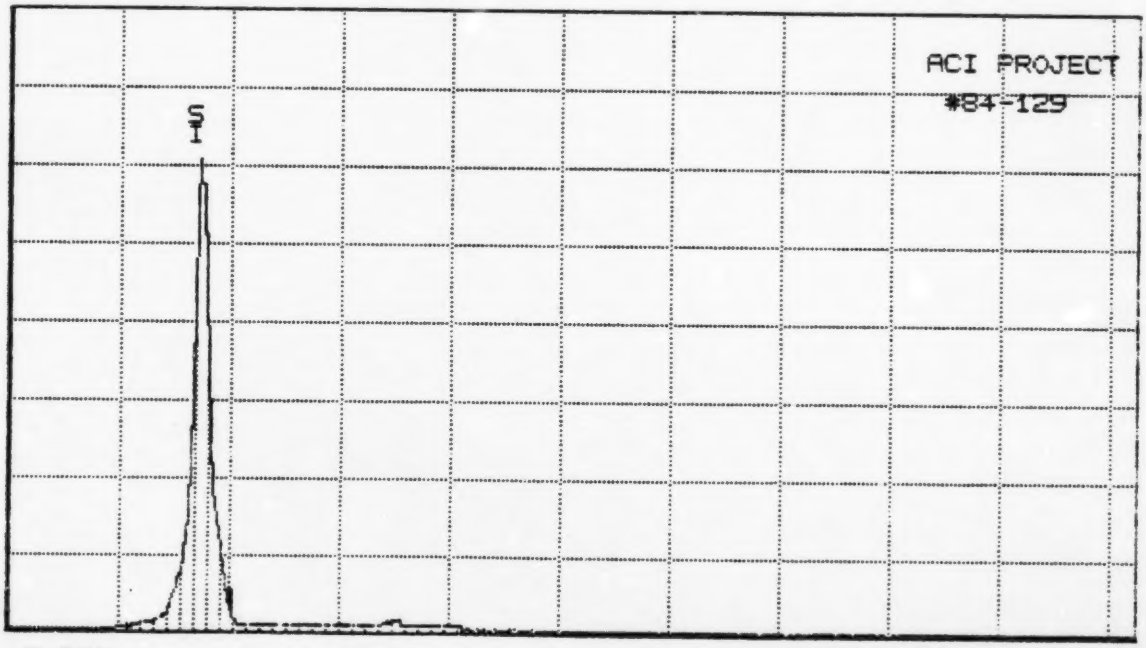
ATLANTIC COAL INSTITUTE TUE 12-MAR-85 17:02
Cursor: 0.000keV = 0



0.000 VFS = 4096 10.240
10 DUST #85-53 20kV MIN ID P#0308-14 spot #6

39

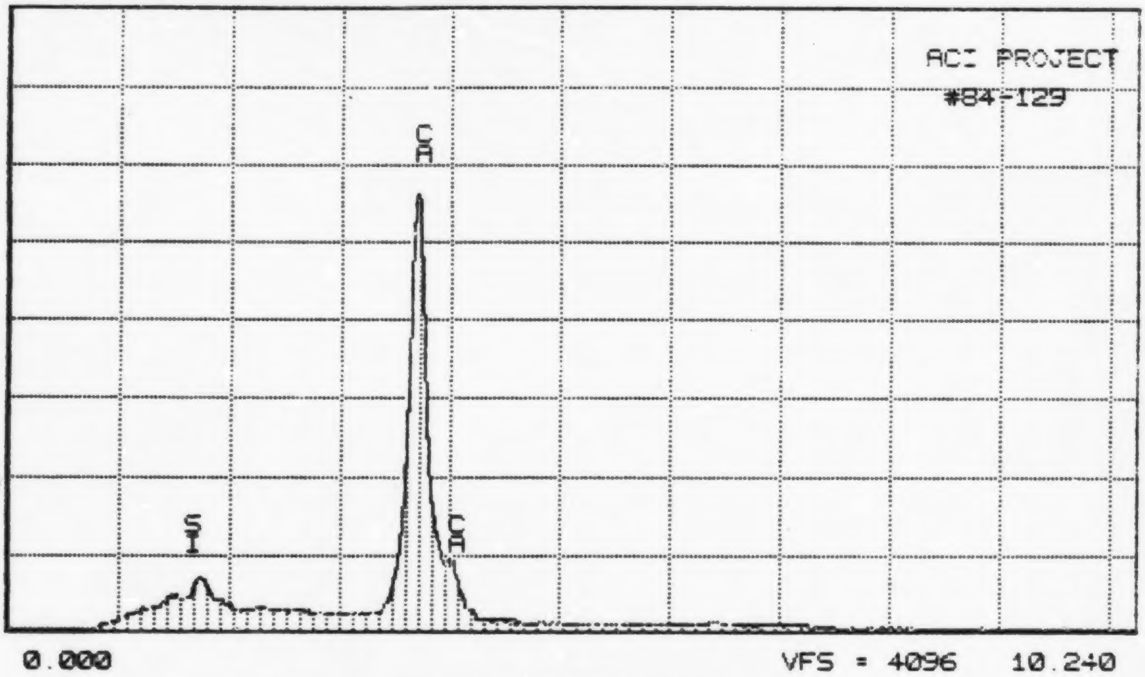
ATLANTIC COAL INSTITUTE TUE 12-MAR-85 17:04
Cursor: 0.000keV = 0



0.000 VFS = 8192 10.240
10 DUST #85-53 20kV MIN ID P#0308-14 spot #7

40

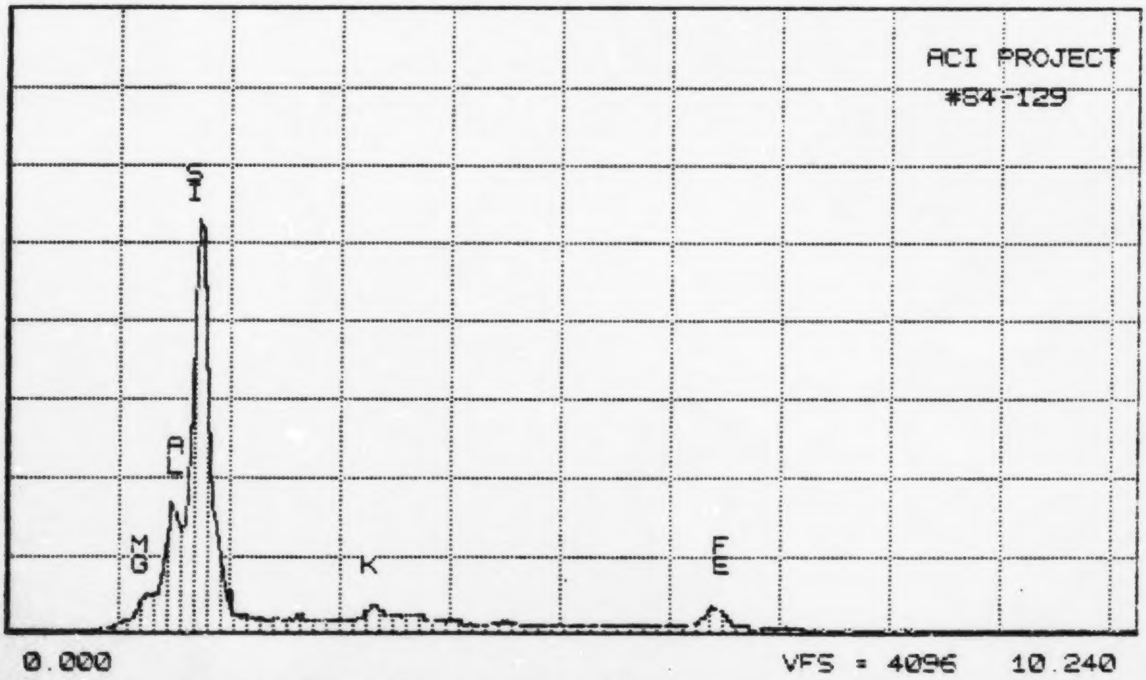
ATLANTIC COAL INSTITUTE TUE 12-MAR-85 17:06
Cursor: 0.000keV = 0



10 DUST #85-53 20kV MIN ID P#0308-14 spot #8

41

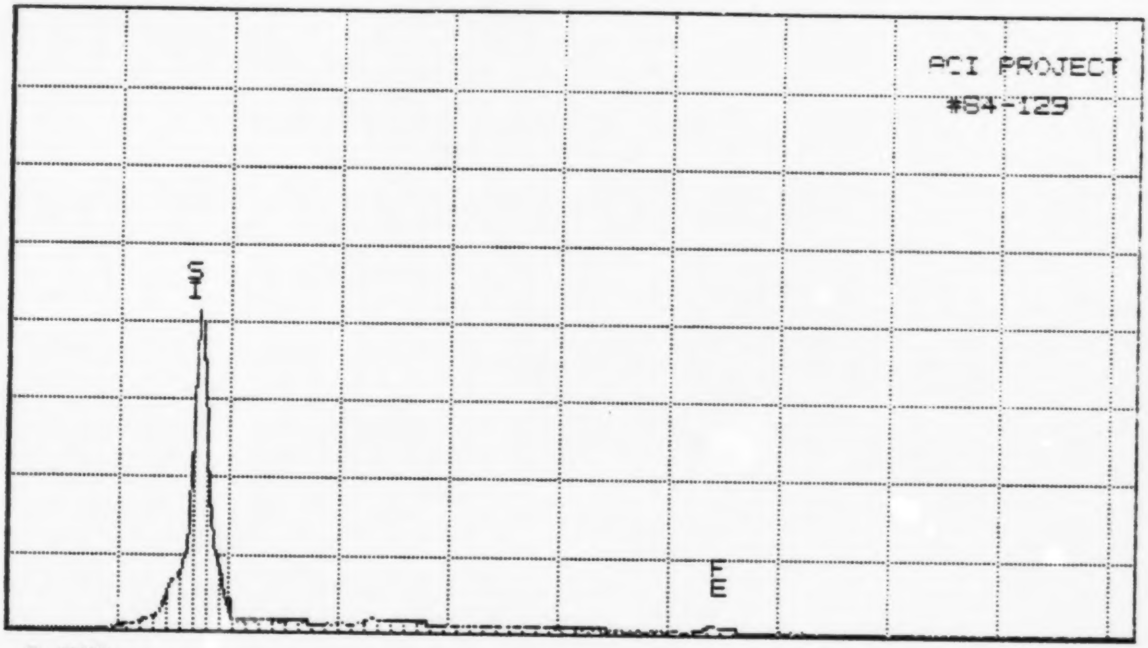
ATLANTIC COAL INSTITUTE TUE 12-MAR-85 17:08
Cursor: 0.000keV = 0



10 DUST #85-53 20kV MIN ID P#0308-14 spot #9

42

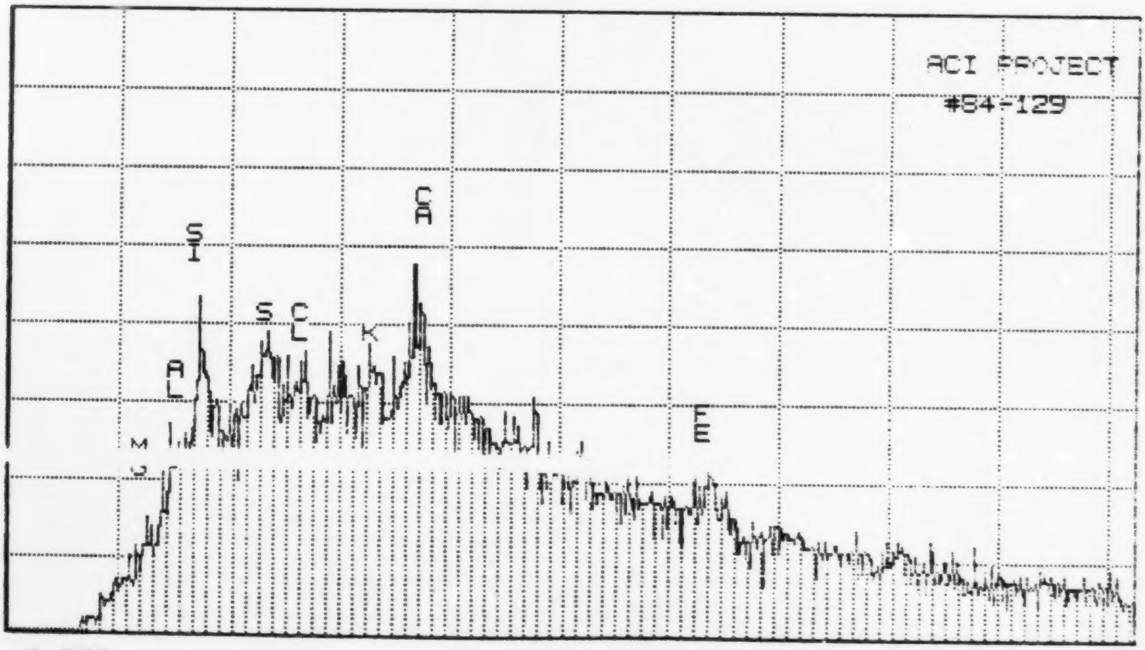
ATLANTIC COAL INSTITUTE TUE 12-MAR-85 17:10
Cursor: 0.000keV = 0



10 DUST #85-53 20kV MIN ID P#0308-14 spot #10

43

ATLANTIC COAL INSTITUTE TUE 12-MAR-85 16:47
Cursor: 0.000keV = 0



10 DUST #85-53 MIN ID 20kV FILTER BACKGND SPOT

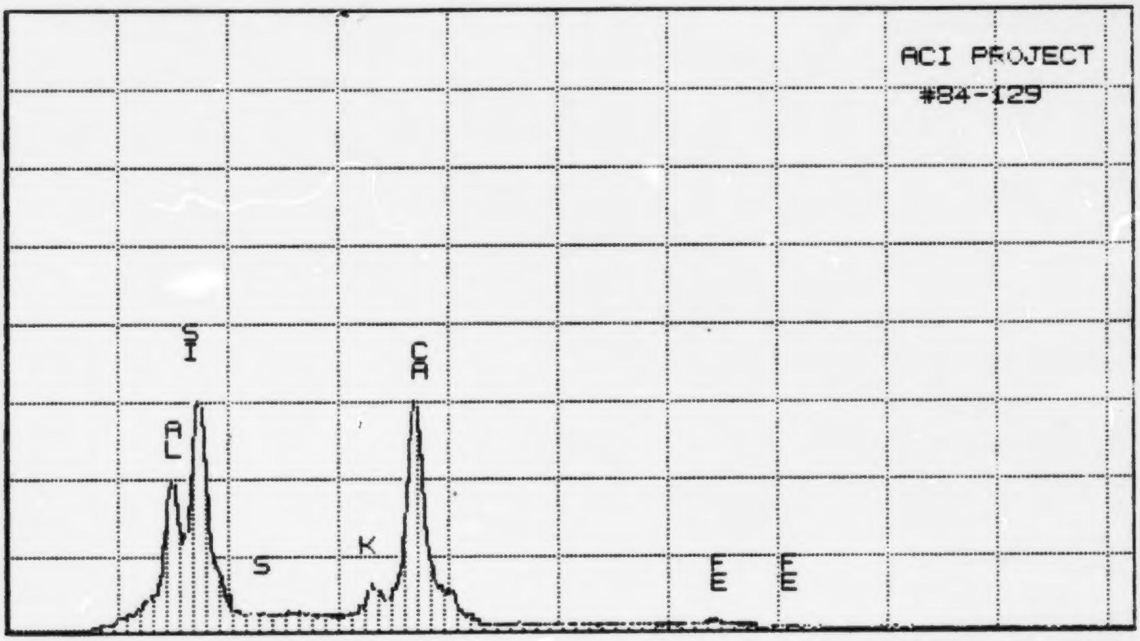
44

7c. MISCELLANEOUS EDX SPECTRA: COAL DUST PARTICULATES WITH UNCOMMON CHEMISTRY

During rapid particulate analysis of coal dust loaded filters, some spectra were not readily classified as one of the common minerals. The following speculative ientifications are offered. Since particle size and geometry was not controlled, some of these traces may be composite spectra due to adjacent grains or due to backscattered electrons:

1. Plagioclase (?)
2. Chlorite (?)
3. Siderite (?)
4. Rutile + Kaolinite or biotite (?)
5. See Appendix II 3c photos 15, 16. An extraneous copper sulphate
6. Rutile
7. Rutile (on gold coated filter)
8. Pyrite (on gold coated filter) with spurious Si peak.
9. Kyanite (?) Al_2SiO_5
10. Unknown iron silicate
11. Illite with associated halite
12. Monazite (?) with associated Kyanite (?)

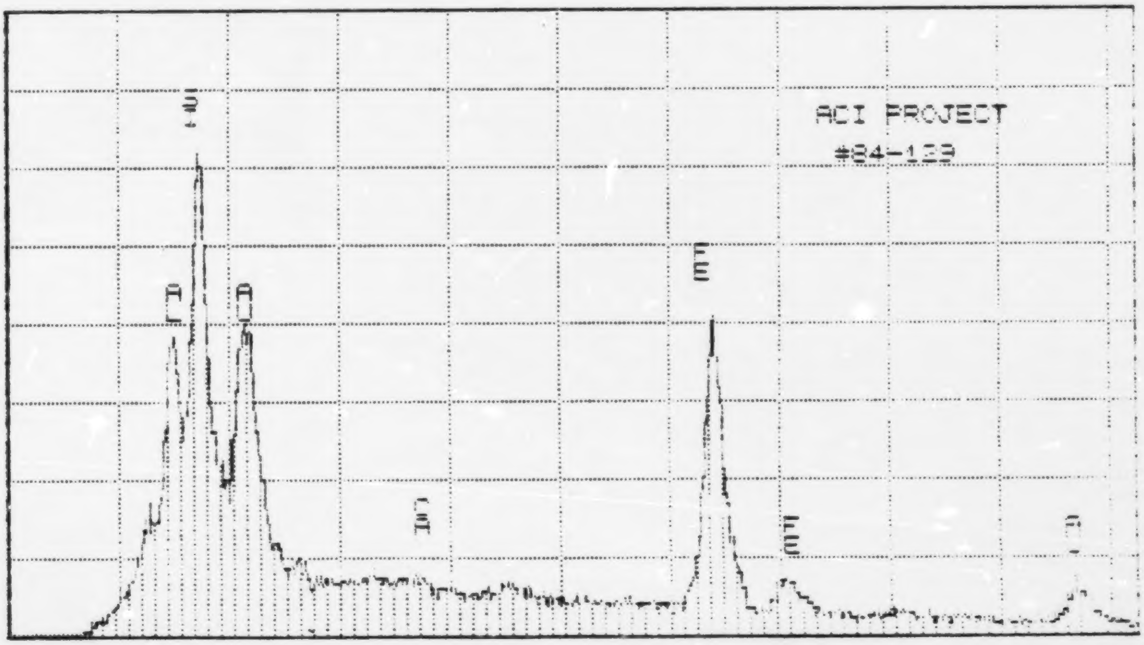
ATLANTIC COAL INSTITUTE THU 07-MAR-85 16:49
Cursor: 0.000keV = 0



0.000 VFS = 2048 10.240
50 DUST #85-38 MIN ID 20 kV SPOT

1

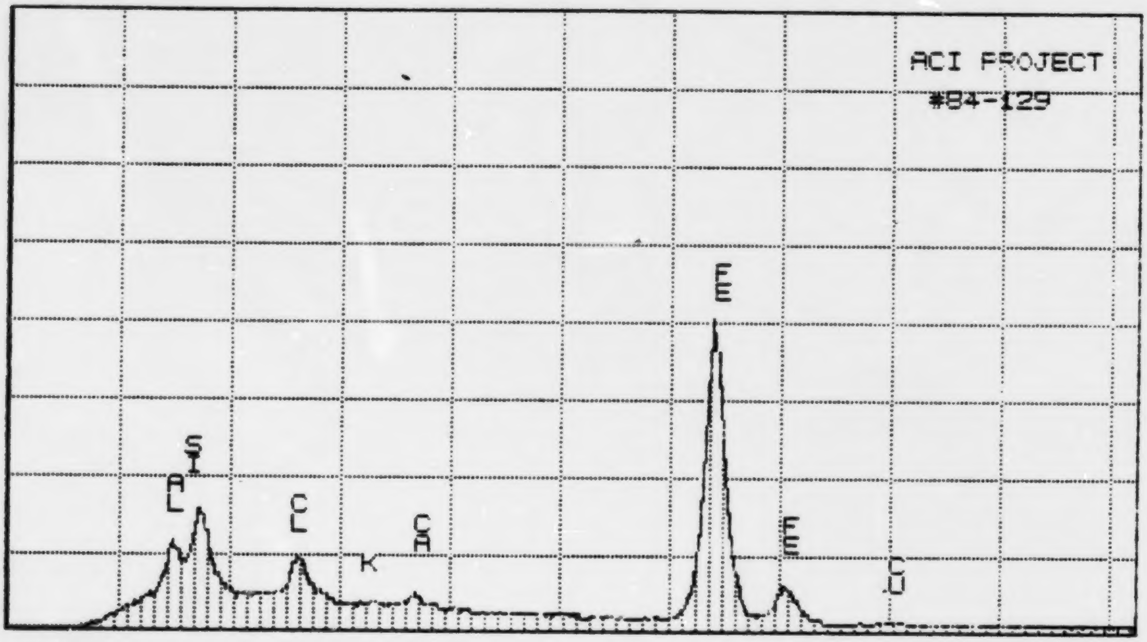
ATLANTIC COAL INSTITUTE WED 10-APR-85 17:06
Cursor: 0.000keV = 0



0.000 VFS = 512 10.240
10 DUST 85-132 PARTICLE

2

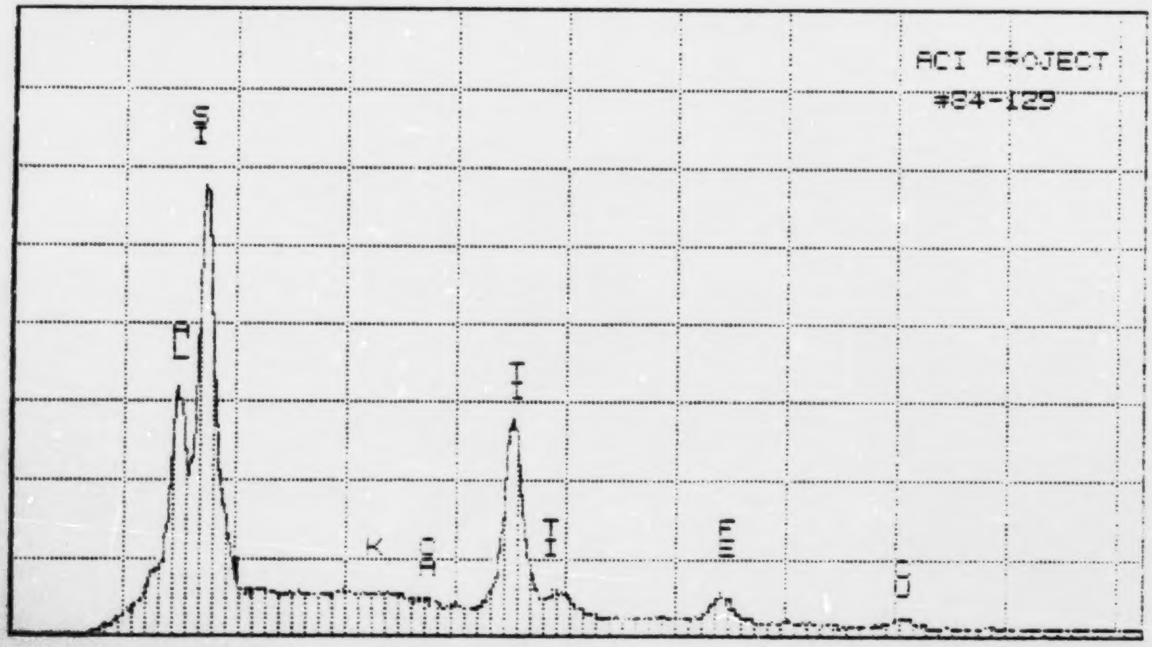
ATLANTIC COAL INSTITUTE MON 18-MAR-85 13:56
Cursor: 0.000keV = 0



3

0.000 VFS = 4096 10.240
10 DUST #85-108 20KV SPOT PARTICLE

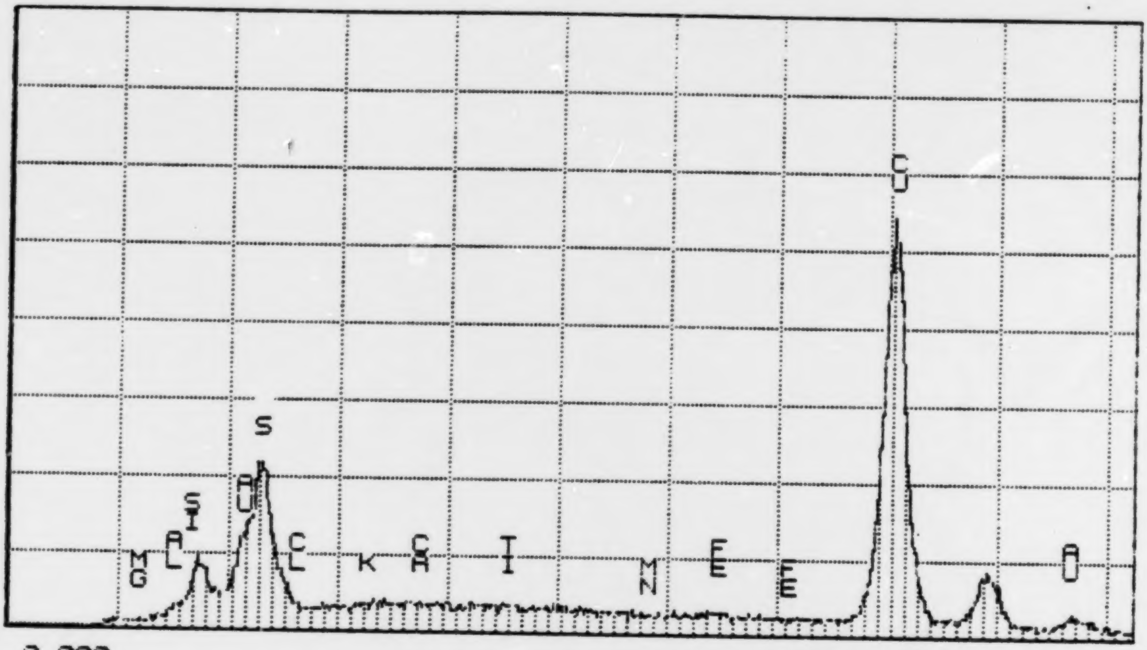
ATLANTIC COAL INSTITUTE WED 27-MAR-85 15:25
Cursor: 0.000keV = 0



4

0.200 VFS = 2048 10.240
9 DUST 85-174 PARTICLE SPOT 20KV

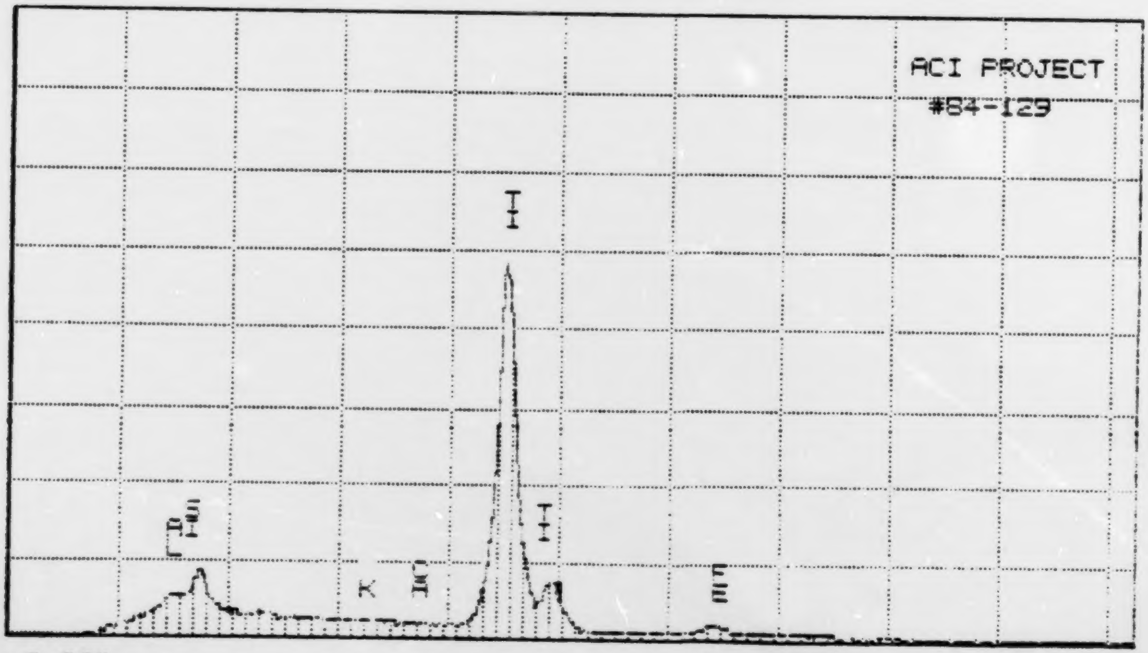
ATLANTIC COAL INSTITUTE FRI 15-MAR-85 16:44
Cursor: 0.000keV = 0



0.000 VFS = 1024 10.240
10 DUST #85-4 20KV P#0314-14 SPOT

5

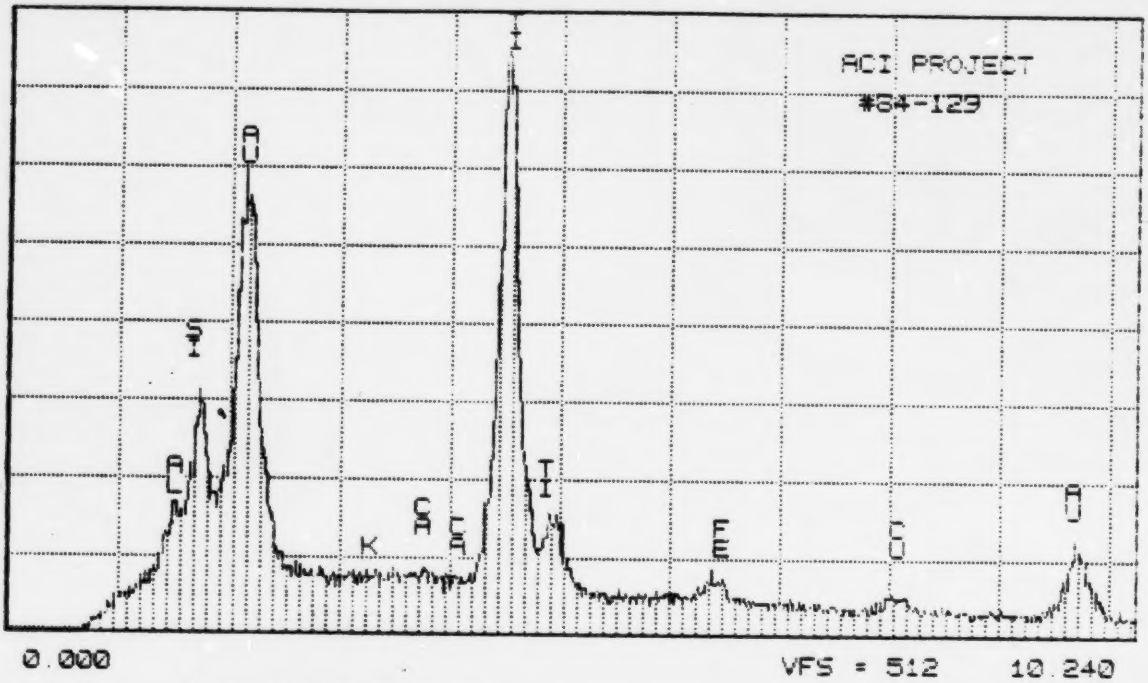
ATLANTIC COAL INSTITUTE WED 27-MAR-85 08:59
Cursor: 0.010keV = 0



0.000 VFS = 4096 10.240
10 DUST 85-175 SPOT PARTICLE 20KV

6

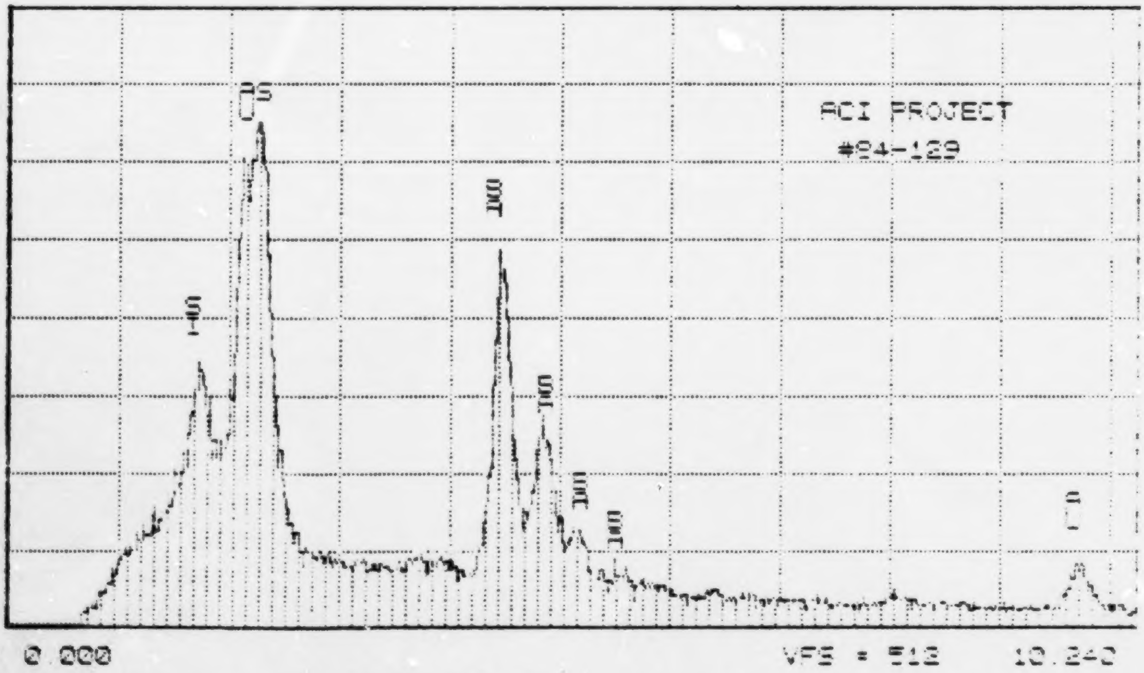
ATLANTIC COAL INSTITUTE WED 03-APR-85 16:02
Cursor: 0.000keV = 0



7 DUST 85-39 PARTICLE

7

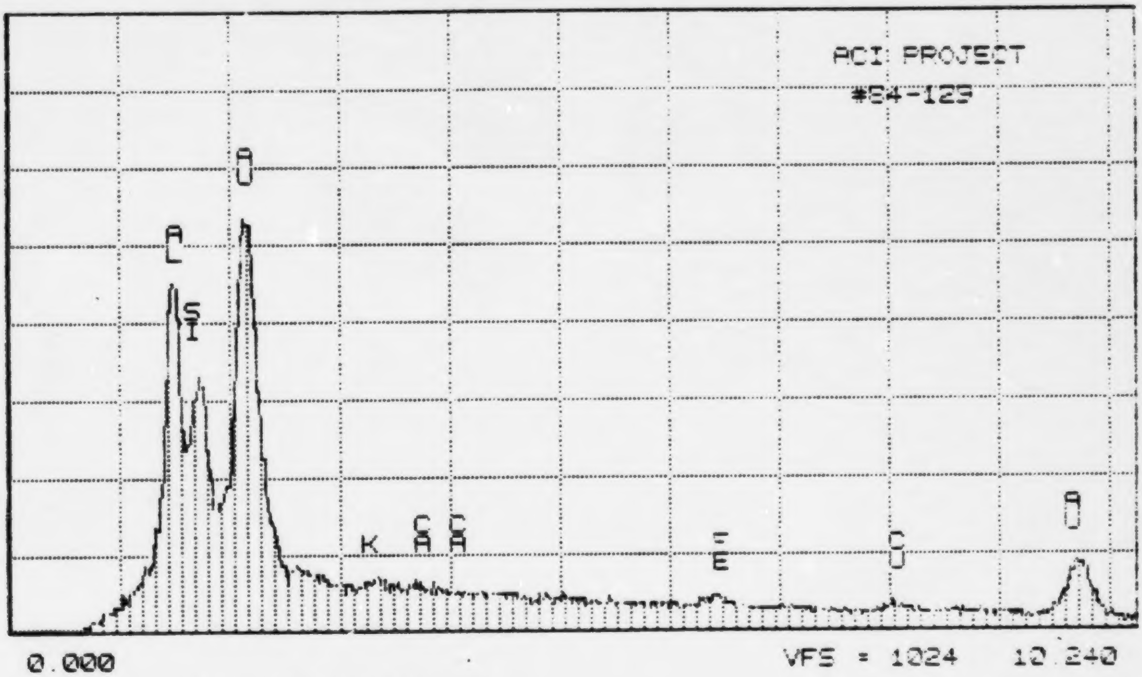
ATLANTIC COAL INSTITUTE WED 10-APR-85 17:28
Cursor: 0.000keV = 0



10 DUST 85-132 PARTICLE

8

ATLANTIC COAL INSTITUTE WED 03-APR-85 15:49
Cursor: 0.000keV = 0

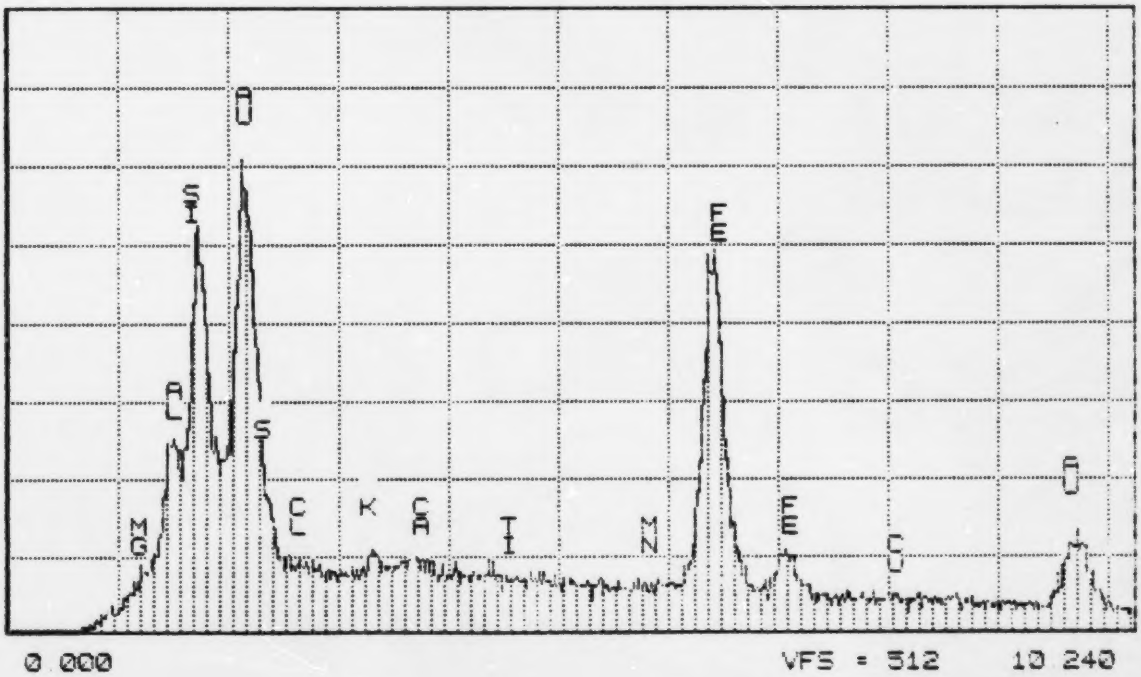


VFS = 1024

9

10 DUST 85-39 PARTICLE

ATLANTIC COAL INSTITUTE FRI 15-MAR-85 16:10
Cursor: 0.000keV = 0



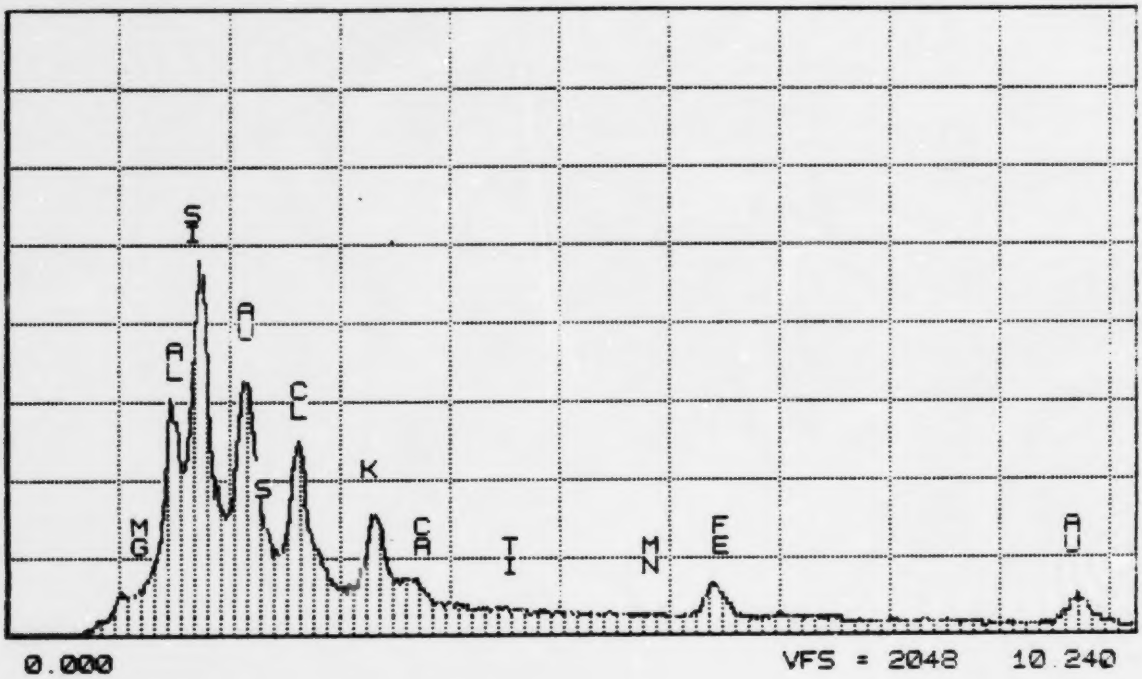
VFS = 512

10

2

ATLANTIC COAL INSTITUTE FRI 15-MAR-85 16:08

Cursor: 0.000keV = 0

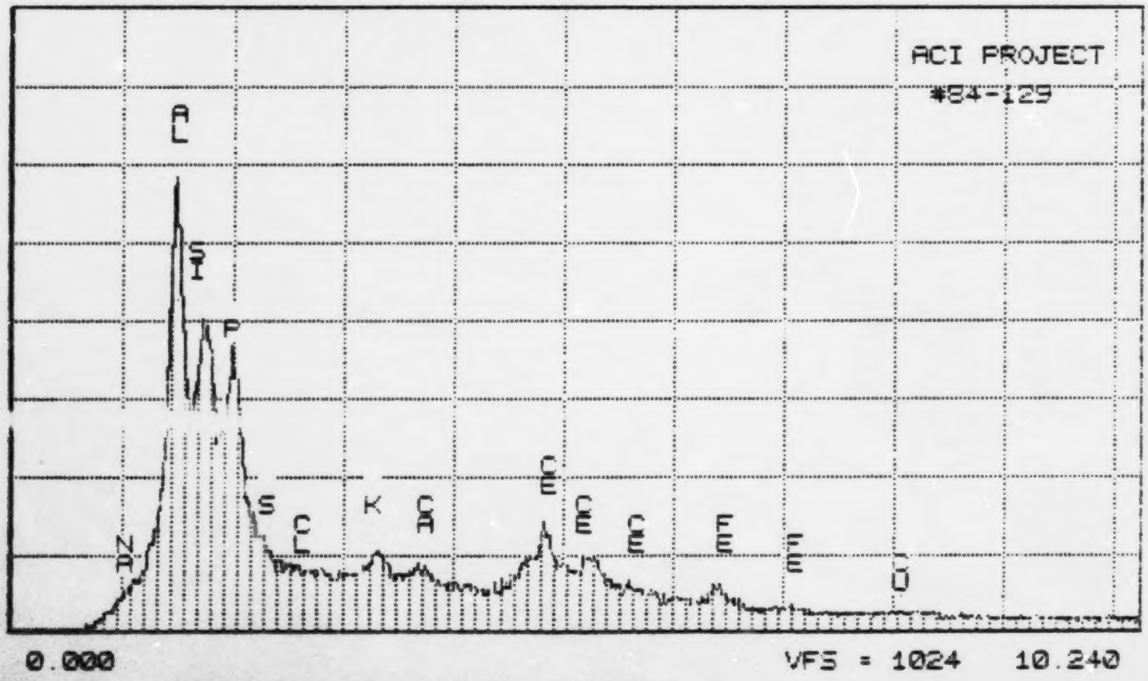


9

11

ATLANTIC COAL INSTITUTE WED 27-MAR-85 08:56

Cursor: 0.010keV = 0



10 DUST 85-175 SPOT PARTICLE 20KV

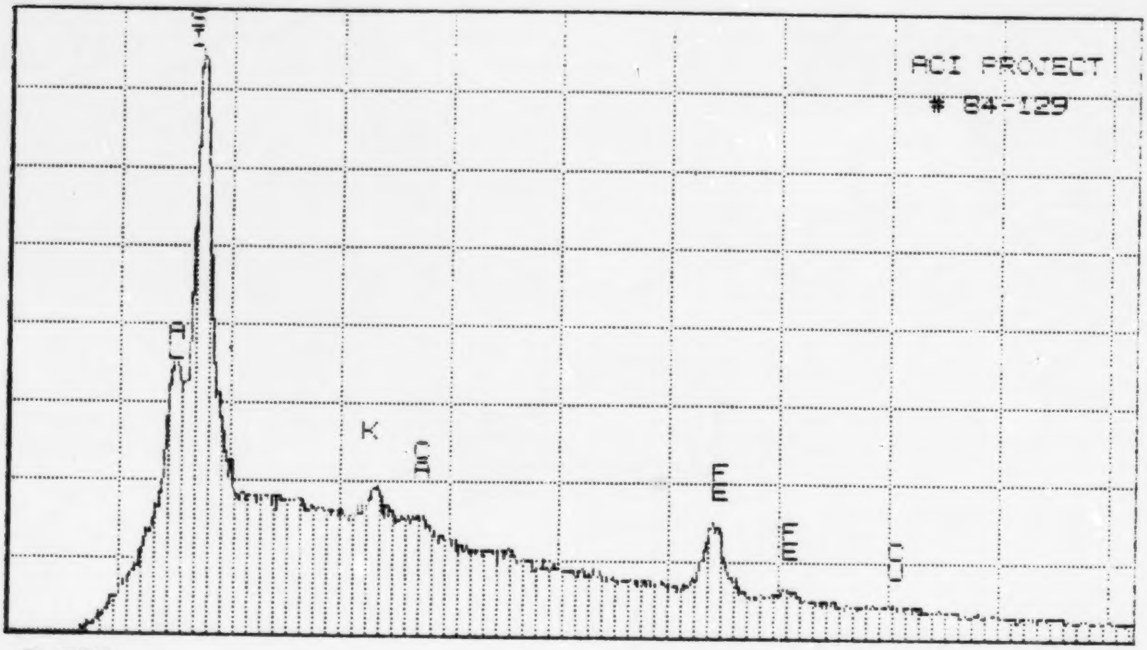
12

8. EDX SPECTRA: FUSED COAL ASH

8a. EDX spectra from three spots on a glass pellet (uncoated) of coal ash fused with lithium tetraborate (sample #85-151-001).. 50 second counts at 20kv. The spectra are essentially identical, indicating homogeneity.
Spectra #1, #2, #3.

8b. Tracor Northern SSQ calculations for standardless semi-semiquantitative analysis: three spot analyses.

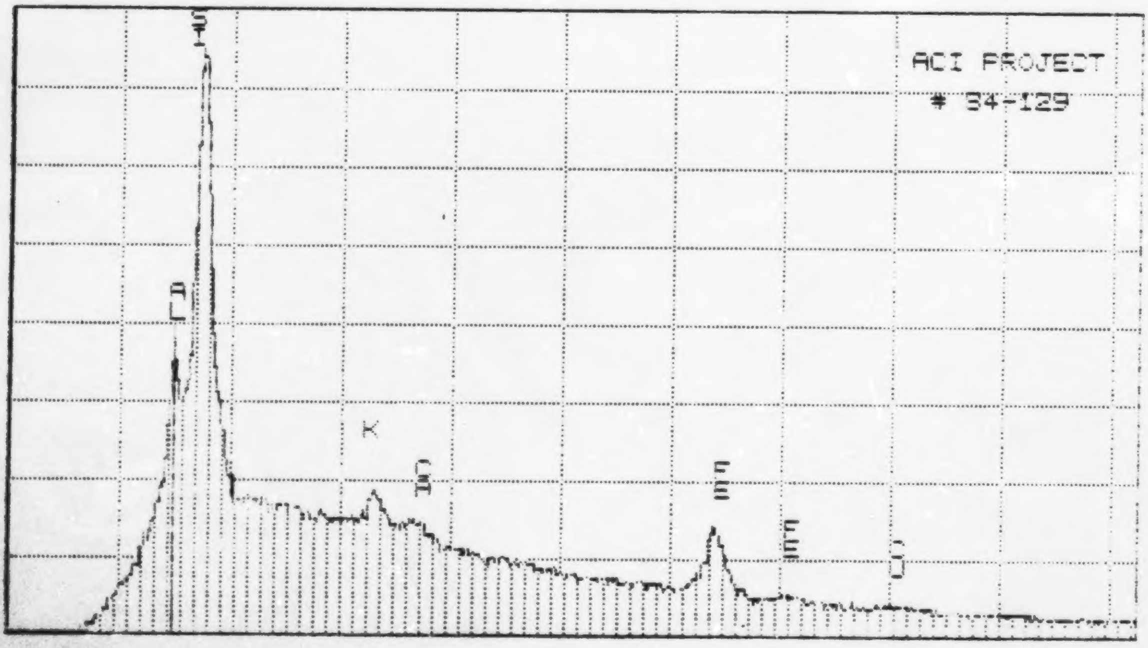
ATLANTIC COAL INSTITUTE MON 25-FEB-85 15:25
Cursor: 0.000keV = 0



0.000 VFS = 2048 10.240
50 FUSED GLASS PELLET, X-RAY ANALYSIS (1)

1

ATLANTIC COAL INSTITUTE MON 25-FEB-85 15:35
Cursor: 0.000keV = 0

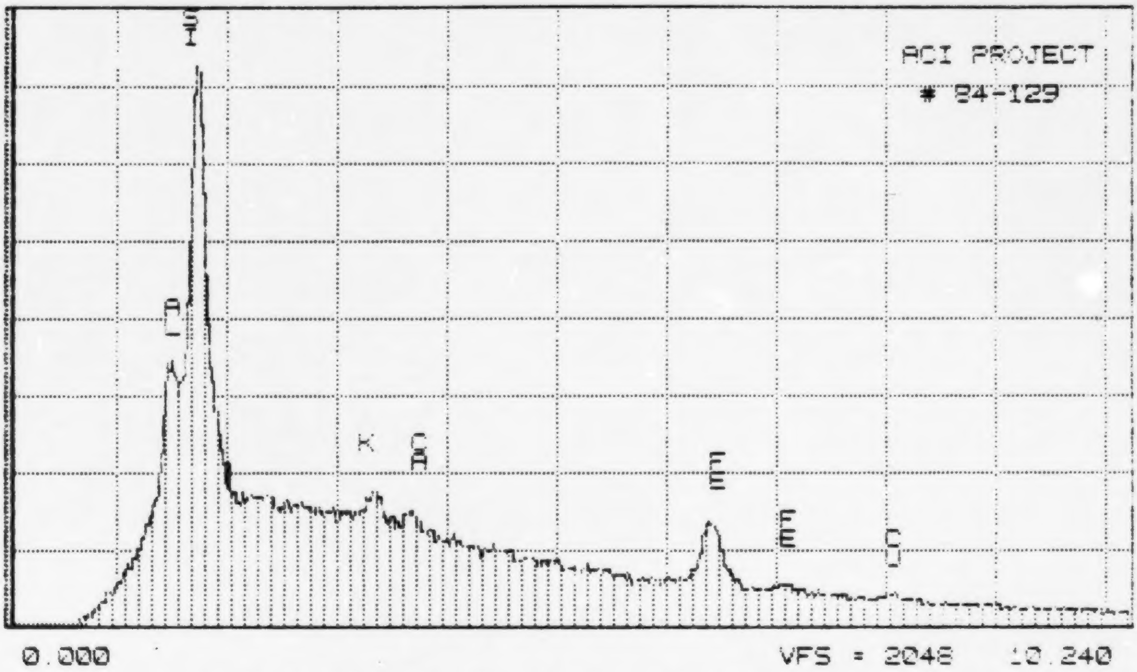


0.000 VFS = 2048 10.240
50 FUSED GLASS PELLET, X-RAY ANALYSIS (2)

2

ATLANTIC COAL INSTITUTE MON 25-FEB-85 16:28

Cursor: 0.070keV = 0



3

50 FUSED GLASS PELLET, X-RAY ANALYSIS (3)

```
SEMI-QUANTITATIVE ANALYSIS: COAL FUSED GLASS PELLET. X-RAY ANALYSIS (1)
        NORM. K-RATIO

AL-K 0.14065 +- 0.00110
S -K 0.50796 +- 0.00199
  -K 0.00613 +- 0.00023
FE-K 0.29404 +- 0.00274
CA-K 0.01986 +- 0.00047
  -K 0.03135 +- 0.00057
CL-K-0.00000 +- 0.00000
TI-K-0.00000 +- 0.00000

 AF CORRECTION        20.00 KV        30.00 Deqs

 .NO.    1

No. of Iterations  7
 ---      K       [Z]     [A]      [F]    [ZAF]   ATOM.%   WT.%
 L-K   0.140   0.972   1.580   0.975   1.499   17.41   14.63   *
SI-K   0.507   0.949   1.676   0.999   1.590   64.24   55.96   *
S -K   0.006   0.969   1.982   0.997   1.918    0.82    0.82   *
 E-K   0.294   1.152   1.023   1.000   1.178   13.80   24.03
 A-K   0.019   1.011   1.223   0.992   1.228    1.36    1.69
K -K   0.031   1.028   1.295   0.993   1.323    2.37    2.88
 L-K   0.000   1.002   1.633   0.995   1.630    0.00    0.00 G *
  -K   0.000   1.117   1.111   0.973   1.209    0.00    0.00 G
 * - High Absorbance

 SQ: SSQ

 EMI-QUANTITATIVE ANALYSIS: COAL FUSED GLASS PELLET. X-RAY ANALYSIS (2)
EL      NORM. K-RATIO

 L-K 0.13805 +- 0.00111
 I-K 0.51000 +- 0.00204
S -K 0.00455 +- 0.00020
 E-K 0.28890 +- 0.00277
 A-K 0.02368 +- 0.00052
K -K 0.03478 +- 0.00062
CL-K-0.00000 +- 0.00000
 I-K-0.00000 +- 0.00000

ZAF CORRECTION        20.00 KV        30.00 Deqs

 T.NO.    2

 o. of Iterations  2
 ---      K       [Z]     [A]      [F]    [ZAF]   ATOM.%   WT.%
AL-K   0.138   0.973   1.571   0.974   1.490   17.03   14.30   *
SI-K   0.510   0.949   1.666   0.999   1.581   64.44   56.11   *
  -K   0.004   0.973   2.001   0.997   1.943    0.62    0.62   *
 E-K   0.288   1.153   1.023   1.000   1.180   13.61   23.71
CA-K   0.023   1.015   1.227   0.992   1.237    1.64    2.04
  -K   0.034   1.031   1.299   0.993   1.332    2.66    3.22
 L-K   0.000   1.013   1.661   0.996   1.677    0.00    0.00 G *
TI-K   0.000   1.129   1.116   0.979   1.234    0.00    0.00 G
 * - High Absorbance
```

```
SQ: SSQ


EMI-QUANTITATIVE ANALYSIS: COAL FUSED GLASS PELLET. X-RAY ANALYSIS (3)
EL     NORM. K-RATIO

L-K 0.13544 +- 0.00111
I-K 0.50318 +- 0.00205
S -K 0.00428 +- 0.00020
E-K 0.30581 +- 0.00289
A-K 0.02329 +- 0.00053
K -K 0.02796 +- 0.00056
CL-K-0.00000 +- 0.00000
I-K-0.00000 +- 0.00000

ZAF CORRECTION        20.00 KV        30.00 Degs

T.NO.   3

o. of Iterations  2
---      K      [Z]     [A]     [F]    [ZAF]   ATOM.%  WT.%
AL-K  0.135  0.971  1.596  0.975  1.512  17.05  14.24  *
SI-K  0.503  0.947  1.681  0.999  1.592  64.18  55.60  *
  -K  0.004  0.970  1.997  0.998  1.934   0.58   0.58  *
FE-K  0.305  1.150  1.022  1.000  1.177  14.44  25.02
CA-K  0.023  1.013  1.222  0.992  1.228   1.61   1.99
  -K  0.027  1.029  1.298  0.992  1.327   2.14   2.58
 L-K  0.000  1.010  1.658  0.996  1.670   0.00   0.00 G *
TI-K  0.000  1.126  1.114  0.977  1.226   0.00   0.00 G
* - High Absorbance




FILE LABEL: PELLETS
AVERAGE W% DATA FOR POINTS   1 TO   3

 POINT    AL-K   SI-K    S -K   FE-K    CA-K    K -K    CL-K    TI-K    TOTAL
   1     14.63  55.96    0.82  24.03    1.69    2.88    0.00    0.00   100.01
   2     14.30  56.11    0.62  23.71    2.04    3.22    0.00    0.00   100.00
   3     14.24  55.60    0.58  25.02    1.99    2.58    0.00    0.00   100.01
         ------ ------ ------ ------ ------ ------ ------ ------   ------
VERAGE   14.39  55.89    0.67  24.25    1.91    2.89    0.00    0.00   100.01
  +/-     0.21   0.26    0.13   0.68    0.19    0.32    0.00    0.00     0.00

COUNT
STATS     0.00   0.00    0.00   0.00    0.00    0.00    0.00    0.00
REL %
```

APPENDIX II

9. Tracor Northern Coal Minerals Programme: test run on coal dust loaded filter for automated particulate recognition and size distribution. Results not available at time of writing.

10. Multielement analysis of coal dust on filters: by instrumental neutron activation analysis (INAA). Results not available at time of writing.

APPENDIX II - 9

PART I

The following pages contain original output from the Tracor Northern TN-5500 x-ray analysis system using "Coal Minerals Analysis" (CMA) software. The sample (large block) is an acrylic lucite coal pellet as described in the body of this report. It should be noted that this analysis represents an illustration of the software only.

```
622-2A/00 CMA LIST







SAMPLE: LARGE BLOCK

NUMBER      1  DISK   100      FILE 101 - 104  PARTICLES                           RUN/T  115.99

RUN         1  DATE  6/ 7/85  SUMMARY   103   TOTAL    352     MASS 3.1E+00  UG/CM2   ANAL./T 55.99

BLOCKS     21  TIME  7:35     OFF-PT. 1      ) 50U     50    ) 50U 2.1E+01  UG/CM2   DEAD/T  60.00

TABLE USED TO SCALE DATA

                          RUN PARAMETER TABLE
  MAG    BIN     XL      XU      PARTS  FIELDS  SCALE     MASS

  750     0      0.2     1.0        6   16.00      40  1.05E-02

  750     1      1.0     2.5        4   16.00      40  1.03E-02

  100     2      2.5     5.0      118    1.03      11  4.04E-01

  100     3      5.0    10.0       81    1.03      11  8.97E-01

   30     4     10.0    20.0       66    1.02       1  2.53E-01

   30     5     20.0    50.0       77    1.02       1  1.46E+00
```

```
SAMPLE: LARGE BLOCK

  MBER     1  DISK   100     FILE 101 - 104  PARTICLES
                                                                              RUN/T  115.99
RUN        1  DATE  6/ 7/85  SUMMARY   103   TOTAL   352    MASS 3.1E+00  UG/CM2  ANAL./T 55.99

  OCKS    21  TIME  7:35     OFF-PT. 1      ) 50U    58   ) 50U 2.1E+01  UG/CM2  DEAD/T  60.00

                                   SIZE DISTRIBUTION

        TYPE            0.2-1   1-2.5   2.5-5   5-10    10-20    +20     PART/CM2   %TOTAL
         QUARTZ...        0       0      32      65       0       3      3.8E+02     0.3
         KAOLINITE        0       0      30      68       1       1      2.9E+03     2.5
         ILLITE...       94       0       0       6       0       0      6.2E+03     5.4
         ORGSULF..        0      39      29      29       1       1      1.2E+03     1.1
         PYJAR....        0       0      80      20       0       0      6.2E+02     0.5
         PYRITE...        0       0      39      55       3       2      1.5E+03     1.4
         FE-SUL...        0       0       0       0     100       0      1.1E+01     0.0
         QUARORG..       99       0       0       1       0       0      5.8E+04    50.6
         QUARPYR..       99       0       0       1       0       0      2.1E+04    18.4
         MIXSIL...        0       0      32      64       2       1      7.7E+02     0.7
         NOT-INT..        5       7      59      23       3       3      2.1E+04    18.7
         UNKNOWN..        0      98       0       0       0       2      4.6E+02     0.4

        TOTAL....        74       2      13       9       1       1      1.1E+05   100.0
```

```
SAMPLE: LARGE BLOCK

NUMBER     1  DISK   100     FILE 101 - 104  PARTICLES                                RUN/T  115.99

RUN        1  DATE  6/ 7/85  SUMMARY   103   TOTAL   352     MASS 3.1E+00  UG/CM2    ANAL./T 55.99

BLOCKS    21  TIME  7:35     OFF-PT. 1      ) 50U     50    ) 50U 2.1E+01  UG/CM2    DEAD/T  60.00

                                  WEIGHT DISTRIBUTION

         TYPE            0.2-1   1-2.5   2.5-5   5-10    10-20    +20   TOTAL

          KAOLINITE         0       0      11      63       2      23     9.2

          ILLITE...         3       0       0      38       0      59     2.3

          ORGSULF..         0       2      22      57       4      14     1.8

          PYRITE...         0       0       9      59       7      25    11.2

          MIXSIL...         0       0      11      56       4      29     2.4

          NOT-INT..         0       0      14      17      10      59    67.8

          MISC.....         4       3      19      46      10      19     5.3

          TOTAL....         0       0      13      30       8      48   100.0
```

```
SAMPLE: LARGE BLOCK

NUMBER     1  DISK   100     FILE 101 - 104  PARTICLES                                    RUN/T  115.99

RUN        1  DATE  6/ 7/85  SUMMARY   103   TOTAL   352     MASS 3.1E+00  UG/CM2   ANAL./T 55.99

BLOCKS    21  TIME  7:35     OFF-PT. 1      > 50U     58    > 50U 2.1E+01  UG/CM2   DEAD/T  60.00

                                      AVERAGE COMPOSITION
         TYPE           AVE      NA    MG    AL    SI     S     CL     P     K     CA    TI    FE   WT%
        QUARTZ...       6.71      0     1     0    94     1      0           1      1     1          1.2
        KAOLINITE       6.89      1          44    47     3      2     0     1      1     2     1    9.2
        ILLITE...       0.87      2     0    35    41     9      0           5      3     3     1    2.3
        ORGSULF..       4.60      1     1     5     2    71      1           1      1     1    18    1.8
        PYJAR....       4.46      1     1     5     5    60      0           3      0     2    23    1.2
        PYRITE...       6.76      0     1     3     2    69                  0      0     2    23   11.2
        FE-SUL...      15.75                  1     5    48                  2            2    42    0.1
        QUARORG..       0.31      0     1    30    30    25      2           5      2     2     4    1.5
        QUARPYR..       0.27                 15    17    40      3           6            1    17    0.8
        MIXSIL...       6.85      1     0    34    54     3      1           2      2     2     2    2.4
        NOT-INT..       5.27      7    10    24    22    20      7     9     9     10    10     8   67.8
        UNKNOWN..       2.68      1     1    14     8    10     16     2     3     18    23     4    0.5
```

APPENDIX II - 9

PART II

The following pages represent original output from the Tracor Northern TN-5500 x-ray analysis system using "Particle Recognition and Characterization" (PRC) and "Stored Image Analysis" (SIA) software. The sample is a nuclepore filter with respirable coal mine dust collected upon it. The analysis is presented as an illustration of the software only.

```
PRC: HISTOGRAM

AVERAGE DIAMETER HISTOGRAM FOR  TYPE:   0   NAME: ALL TYPES
BIN MAX(uM)    NO.  %   00---10---20---30---40---50---60---70---80---90---100
     0.33       0   0.0 [
     0.67       0   0.0 [
     1.00       0   0.0 [
     1.33       0   0.0 [
     1.67       1   5.8 [***
     2.00       3  17.6 [*********
     2.33       2  11.7 [******
     2.67       2  11.7 [******
     3.00       2  11.7 [******
     3.33       2  11.7 [******
     3.67       3  17.6 [*********
     4.00       0   0.0 [
     4.33       0   0.0 [
     4.67       0   0.0 [
     5.00       1   5.8 [***
     5.33       0   0.0 [
     5.67       1   5.8 [***
     6.00       0   0.0 [

AVERAGE DIAMETER HISTOGRAM FOR  TYPE:  63   NAME: UNKNOWN
BIN MAX(uM)    NO.  %   00---10---20---30---40---50---60---70---80---90---100
     0.33       0   0.0 [
     0.67       0   0.0 [
     1.00       0   0.0 [
     1.33       0   0.0 [
     1.67       1   5.8 [***
     2.00       3  17.6 [*********
     2.33       2  11.7 [******
     2.67       2  11.7 [******
     3.00       2  11.7 [******
     3.33       2  11.7 [******
     3.67       3  17.6 [*********
     4.00       0   0.0 [
     4.33       0   0.0 [
     4.67       0   0.0 [
     5.00       1   5.8 [***
     5.33       0   0.0 [
     5.67       1   5.8 [***
     6.00       0   0.0 [
SIA-1B/80 .MAIN

PARTICLE ANALYSIS SUMMARY
LABEL:ACI
NUMBER OF FRAMES:          1
NUMBER OF PARTICLES:      96
MAGNIFICATION:           700
FIELD WIDTH:        142.85 UM
FIELD AREA:       20408.16 UM^2

AVERAGE PARAMETER VALUES FOR LAST  96 PARTICLES

AVE DIAM. (UM)          4.98      +/-       3.52

AREA (UM^2)            12.42      +/-      12.30

PERIMETER (UM)         18.82      +/-      21.69

X-FERET (UM)            4.96      +/-       2.39

Y-FERET (UM)            4.77      +/-       2.75

LENGTH (UM)             6.49      +/-       4.69

WIDTH (UM)              3.77      +/-       2.21

ORIEN.ANG (DEG)        56.51      +/-      56.18
```

PARTICLE LISTING

PART.	AVE.DIAM (UM)	X-FERET (UM)	Y-FERET (UM)	AREA (UM^2)	PERIM (UM)	CNTR-X (UM)	CNTR-Y (UM)	LENGTH (UM)	WIDTH (UM)	ORIEN DEG
1	2.79	3.90	2.23	4.98	7.81	125.04	3.90	3.90	2.23	0
2	6.13	7.81	4.46	19.61	21.76	45.20	3.34	7.81	4.46	0
3	8.37	5.02	3.90	8.71	13.95	126.67	6.69	12.27	3.90	0
4	2.79	2.79	2.79	4.35	6.69	133.27	9.48	3.34	2.79	146
5	5.02	6.13	3.90	13.70	15.06	67.52	11.71	6.13	3.90	0
6	2.23	2.23	2.79	3.42	6.13	125.04	12.83	2.79	2.23	11
7	3.34	4.46	2.79	6.53	9.48	140.06	13.39	4.46	2.79	0
8	2.79	3.34	2.79	3.11	17.85	117.18	16.74	3.34	1.67	0
9	4.46	3.90	5.02	10.58	10.95	56.36	16.18	5.58	3.90	112
10	3.90	3.90	4.46	7.16	10.60	110.49	16.18	5.02	2.79	157
11	5.58	6.13	6.13	14.01	19.53	129.46	16.18	6.13	5.58	0
12	5.02	6.69	3.90	14.63	17.29	120.53	20.64	6.69	3.90	0
13	3.90	5.02	3.34	6.85	12.83	135.04	21.76	5.58	3.34	168
14	11.16	9.48	13.95	50.13	34.04	101.88	18.41	14.50	6.69	78
15	2.23	2.23	2.79	3.42	6.13	120.98	24.55	2.79	2.23	33
16	3.90	5.02	3.90	9.65	10.41	125.55	24.55	5.02	3.90	0
17	2.23	2.79	2.23	3.11	5.58	44.88	26.78	2.79	2.23	0
18	2.79	2.79	2.79	4.35	6.69	137.27	27.34	3.34	2.23	101
19	22.87	13.95	14.50	56.34	53.01	110.49	23.99	30.69	13.39	11
20	7.25	7.81	7.25	20.55	22.87	31.89	28.45	9.48	4.46	123
21	2.79	2.79	2.79	4.35	6.69	119.41	32.36	3.34	2.23	157
22	3.90	3.90	4.46	10.27	10.60	140.06	31.24	5.02	3.90	101
23	3.90	5.02	3.34	7.16	9.48	88.16	34.59	5.58	2.79	157
24	2.79	2.79	3.34	4.67	7.25	110.86	37.38	3.90	2.23	67
25	2.79	2.79	3.34	4.98	7.81	100.81	37.94	3.90	2.79	67
26	3.34	3.90	3.34	6.85	8.92	123.88	37.94	4.46	2.79	168
27	5.02	5.02	5.58	16.50	15.06	113.20	38.50	6.13	5.02	112
28	5.02	4.46	6.13	10.27	15.06	45.20	39.06	6.69	3.90	101
29	2.79	3.34	2.79	5.60	7.81	82.50	41.85	3.90	2.79	11
30	3.34	3.90	3.34	5.60	8.92	30.69	42.41	3.90	2.79	0
31	2.79	2.79	3.34	4.35	7.25	134.48	42.41	3.90	2.23	112
32	3.34	2.79	4.46	6.85	10.04	139.58	42.41	4.46	2.79	67
33	2.79	3.34	2.79	5.29	7.25	116.07	44.08	3.90	2.23	11
34	2.79	3.34	2.79	4.98	7.25	126.67	46.31	3.90	2.23	22
35	18.97	7.81	6.69	18.68	105.46	121.65	44.64	27.34	6.69	11
36	3.34	3.34	3.90	7.16	9.48	21.76	46.87	4.46	2.79	101
37	6.69	6.13	7.81	18.16	21.20	134.48	49.10	8.92	3.90	45
38	3.34	3.90	3.34	4.98	10.60	119.97	54.68	4.46	2.79	11
39	5.02	3.34	6.69	13.07	16.18	135.60	55.24	7.25	3.34	78
40	5.58	5.02	7.25	17.43	16.74	45.73	57.47	7.25	5.02	78
41	4.46	5.58	3.79	9.96	11.71	32.36	62.49	6.13	2.79	22
42	2.79	3.34	2.79	4.98	7.25	47.43	65.29	3.90	2.79	157
43	4.46	3.90	5.58	9.83	17.85	41.29	65.29	5.58	3.90	56
44	16.74	11.71	16.18	66.32	136.16	120.34	68.26	18.41	15.06	112
45	7.25	8.37	6.13	28.02	42.49	116.62	65.84	8.92	5.58	11
46	4.46	4.46	4.46	11.52	12.83	71.42	68.63	5.58	3.34	146
47	2.23	2.79	2.23	3.11	5.58	137.27	70.31	3.34	1.67	11
48	2.79	3.90	2.79	5.60	8.37	13.95	70.87	3.90	2.79	0
49	2.79	3.34	3.34	5.60	8.37	122.76	70.87	3.34	2.79	0
50	5.02	6.13	3.90	11.52	15.62	69.75	72.54	6.13	3.90	0
51	5.58	6.69	5.02	15.57	43.61	112.72	72.54	7.25	5.02	146
52	3.34	3.34	3.90	4.67	10.04	119.41	74.21	4.46	2.23	45
53	3.34	3.90	3.34	6.22	8.92	132.81	74.21	4.46	2.23	112
54	3.34	2.23	3.90	5.29	8.37	68.63	76.45	3.90	2.23	56
55	15.62	9.48	7.25	30.82	34.04	116.07	79.24	22.87	6.69	11
56	4.46	4.46	4.46	10.89	11.71	135.60	80.37	5.02	3.34	11
57	9.48	6.69	12.27	28.64	35.71	130.82	82.50	12.83	5.58	112
58	7.81	8.37	8.37	28.96	36.83	120.33	84.49	9.48	7.25	135
59	2.23	2.79	2.23	3.11	5.58	86.49	93.19	2.79	2.23	0
60	5.02	5.58	5.02	10.89	14.50	133.92	93.19	6.69	3.34	22
61	2.79	3.90	2.23	5.60	7.81	82.50	94.86	4.46	2.23	168
62	11.71	12.83	11.16	63.82	88.91	113.31	93.19	13.39	11.16	11
63	4.46	5.58	3.90	9.96	13.95	90.77	101.80	6.13	3.34	11
64	2.79	3.34	2.79	4.98	7.25	125.04	103.23	3.90	2.79	22
65	6.13	6.69	6.69	14.94	18.41	40.73	102.12	7.81	5.02	33
66	2.23	2.23	2.79	3.42	6.13	30.03	108.01	3.34	2.23	78
67	9.48	10.60	8.92	30.80	97.09	126.11	106.02	11.71	7.81	157
68	6.69	6.69	7.25	25.24	21.76	117.74	108.01	8.92	4.46	33
69	4.46	5.58	4.46	9.96	15.06	39.06	110.49	5.58	3.90	0
70	5.58	4.46	6.69	16.19	15.62	121.09	112.72	7.25	3.90	78
71	5.58	5.02	6.69	13.08	16.18	137.27	112.72	6.69	4.46	78
72	4.46	6.13	3.90	13.70	13.95	23.43	115.31	6.13	3.90	0
73	2.79	2.23	3.34	3.73	7.81	23.43	119.97	3.34	2.23	45
74	2.23	3.34	2.23	3.42	6.69	93.19	120.53	3.34	1.67	0
75	2.79	3.34	2.23	3.42	6.69	109.93	122.20	3.90	1.67	11
76	5.02	5.58	5.58	14.94	15.06	115.31	121.09	6.13	3.90	101
77	10.04	9.48	11.16	39.54	40.73	131.13	118.30	11.71	8.92	67
78	3.90	5.02	3.34	8.09	11.71	89.04	122.76	5.58	2.79	22
79	4.46	5.02	3.90	13.07	13.39	121.09	123.88	5.58	3.90	146
80	6.69	6.69	8.37	17.12	26.78	17.29	122.76	9.48	3.90	45
81	4.46	5.58	3.34	12.45	14.50	73.10	124.99	6.13	3.34	168
82	5.02	6.13	4.46	8.71	14.50	92.07	126.67	6.69	2.79	11
83	3.90	3.90	3.90	8.09	10.60	29.00	127.79	4.46	3.34	67
84	3.34	3.34	3.34	7.47	8.92	97.65	130.30	3.90	3.34	45
85	2.79	3.34	3.34	5.29	7.81	119.41	130.30	3.90	2.79	11
86	2.23	2.79	2.23	3.73	6.13	124.44	131.13	3.34	2.23	157
87	4.46	4.46	5.02	9.96	13.39	23.43	130.58	5.58	3.34	33
88	2.79	2.79	3.34	4.04	7.25	137.27	131.69	3.34	2.23	11
89	2.79	2.79	3.34	4.98	7.25	116.62	132.81	3.90	2.79	78
90	7.25	8.37	7.25	20.86	22.87	84.47	132.81	9.48	5.02	11
91	3.90	5.02	3.34	8.71	29.01	127.79	134.48	5.02	3.34	0
92	2.23	2.79	2.79	3.73	6.13	106.02	136.71	2.79	2.23	0
93	6.13	8.37	3.90	15.25	41.85	130.04	136.16	8.37	3.90	0
94	4.46	5.02	5.02	12.45	12.83	26.83	137.27	5.02	3.90	0
95	3.34	2.79	3.90	4.04	9.48	125.33	137.83	4.46	2.23	67
96	2.79	2.79	2.79	4.04	6.69	113.72	138.95	3.34	2.79	78

APPENDIX II - 10

The following pages contain the data from Neutron Activation analysis of respirable dust samples, R.O.M. coal, and stone dust. Appendix II-10a is a tabular summary for all of the INAA data. The data received from Neutron Activation Services shows good correlation to values reported by the National Bureau of Standards for NBS coal Standard SRM-2685. The data reported for filter samples from five different sites show no obvious trends or any significant deviation. Most notably, the concentration of gold reported for sample 85-38 is a full order of magnitude higher than all other samples but within the ppb range. The values reported for a bare Millipore filter indicate little, if any, elemental contribution to the respirable dust samples from the filter.

The most likely source of a given element present in a respirable dust sample can be determined by comparing data for the coal, stone dust and bare filter. As most elements are below detectable limits in the filter material, the filter is not considered a source. Where an elemental concentration in the Run-of-Mine coal exceeds that in the stone dust by greater than an order of magnitude; and the respirable dust concentration is greater for that element than that for stone dust; the most likely source for that element in the respirable dust sample is the coal or host rock. This is the case for Na, As, Ba, Br, Cl, Ce, Co, Cr, Pb. Conversely, the most likely source of Ca is the stone dust. Unfortunately, due to lack of separate samples for "pure" coal, and roof rock; a distinction of their elemental contributions to the respirable dust cannot be made.

The INAA results for the 18 respirable dust samples analysed provide little significant comparative information. More detailed Neutron Activation analyses should be carried out and include a preconcentration step to increase elemental sensitivity. Also whole coal and rock separates from a Run-of-Mine coal sample should be included.

NEUTRON ACTIVATION ANALYSIS DATA

ELEMENT	DETECTION LIMIT	NAS VALUES COAL STD. SRM-2685	CERTIFIED NBS DATA SRM-2685	SYDNEY COAL LINGAN-HARBOR	CBDC STONE DUST	MILLIPORE FILTER	WALL FACE (avg.)	BRUSHER TOP (avg.)	BRUSHER BOTTOM (avg.)	CONTROL POINT (avg.)	GENERAL SAMPLE (avg.)
Al	0.0010 %		1.7 %		0.68 %	0.001 %	0.19 %	0.19 %	0.09 %	0.18 %	0.15 %
Ca	0.0500 %	0.3 %	0.52 %	<0.1 %	65.2 %	<0.05 %	<0.1 %	0.15 %	* 0.13 %	0.10 %	<0.1 %
Fe	0.0010 %	2.3 %	2.9 %	2.2 %	0.32 %	0.002 %	0.13 %	0.20 %	0.08 %	0.10 %	0.11 %
Mg	0.0500 %		0.1 %	0.25 %	0.58 %	* <0.05 %	<0.05 %	<0.05 %	<0.05 %	<0.05 %	<0.05 %
Na	1.0000	680	0.08 %	0.12 %	25	16	238.33	225.00	230.00	310.00	240.00
Ti	5.0000		0.09 %		0.02 %	<5	130.00	130.00	94.00	120.00	116.00
Ag	0.1000				2.5		0.95	0.50	1.15	0.50	0.25
As	0.0500	12	12	36	0.28	<0.05	1.03	0.75	0.95	1.60	0.95
Au	1.0000 ppb	31 ppb		2 ppb	<5 ppb	6 ppb	17.17 ppb	141.00 ppb	24.50 ppb	25.00 ppb	21.50 ppb
Ba	10.0000	90	105	210	40	<10	33.33	50.00	45.00	60.00	50.00
Be	1.0000				<1						
Br	0.5000	5	5.6	11	0.41	1	12.10	12.20	19.00	17.00	17.50
Cd	0.2000				<0.2						
Ce	0.5000	16	18	31.2	1	<0.1	2.67	2.15	1.40	2.20	2.10
Cl	10.0000			980	60	30	445.00	430.00	455.00	480.00	505.00
Co	0.0100	4.3	4.6	8	0.21	0.05	1.22	1.25	1.50	2.00	1.40
Cr	0.1000	21	22	35	1.2	0.6	7.97	9.65	7.25	9.20	8.25
Cs	0.0500	1.5	1.3	3.7	0.08	<0.05	0.42	0.65	0.75	0.50	1.75
Cu	0.0500				3.5						
Dy	0.0050			2.1	0.492	<0.005	0.11	0.11	0.05	0.10	0.07
Ga	1.0000			10	2	<1	8.17	9.50	11.00	12.00	9.50
Hf	0.0100	0.9	0.91	1.1	0.09	0.01	0.17	* 0.75	0.20	0.30	0.15
I	0.1000			<10	3	0.1	3.82	3.55	4.50	3.90	3.00
In	5.0000 ppb				240 ppb	<5 ppb	* 16.0 ppb	26.00 ppb	45.00 ppb	110.00 ppb	39.00 ppb
K	500.00		0.26 %	1.1 %	0.19 %	<500	383.33	* 900	* 375	600.00	<500
La	0.0010	8.6	10	16.6	0.482	0.005	1.08	1.00	0.50	0.90	0.75
Lu	0.0050	0.12		0.16	0.006	<0.005	* 0.04	0.01	<0.01	0.01	0.01
Mn	0.1000		41	230	550	0.3	20.67	19.50	14.50	19.00	17.00
Mo	0.2000	1.2		1.8	0.2	<0.02	* 0.88	2.45	1.05	0.90	* 1.2
Nd	0.5000	7			<3	<3	* 0.6	* 0.5	<0.5	<0.5	<0.5
Ni	5.0000	21		41	15	<5	<50.00	<50.00	<50.00	<50.00	<50.00
Pb	0.2000				50						
Rb	0.5000	17	17	58	1.1	<0.5	* 2.5	* 1.5	* 0.05	<2.00	* 2.75
Sb	0.0050	0.3	0.36	1.4	0.075	0.012	2.25	1.35	3.05	1.70	2.35
Sc	0.0050	3.7	3.7	6.7	0.17	<0.005	0.44	0.46	0.23	0.41	0.35
Se	0.1000	1.6	1.9	2.1	<0.1	<0.1	* 0.45	<0.50	<0.50	<0.50	<0.50
Sm	0.0050	1.43	1.7	2.55	0.103	<0.005	0.17	0.16	0.08	0.14	0.13
Sr	10.0000	80		<50	50	<10	<50.00	<50.00	<50.00	<50.00	<50.00
Ta	0.0500	0.2		0.2	<0.05	<0.05	* 0.11	<0.1	* 0.13	<0.1	<0.1
Tb	0.0500	0.2		0.37	<0.1	<0.1	<0.05	<0.05	<0.05	<0.05	<0.05
Th	0.0100	2.4	2.7	4.4	0.11	<0.01	0.27	0.30	0.15	0.20	0.25
U	0.0050	0.91	0.95	1.08	0.517	<0.005	0.11	0.10	0.17	0.07	0.10
V	0.1000		31	47	7.1	0.8	14.67	17.00	19.00	21.00	16.50
W	0.5000	1	1.2	1	<0.5	<0.5	<1.00	<1.00	<1.00	<1.00	<1.00
Yb	0.0050	0.7		0.96	0.041	<0.005	0.06	0.07	0.02	0.06	0.04
Zn	1.0000	25	17	65	14	1	53.00	62.50	52.00	91.00	89.50

NOTES:

1. All values reported as ppm unless otherwise noted
2. Values reported for samples collected and analysed on filters are not corrected for filter contribution.

APPENDIX II - 10b

Neutron Activation (Raw Data)

The sample numbers assigned by the Atlantic Coal Institute and their corresponding descriptions are listed below for raw data given in the pages to follow. Samples were sent January 21, 1985. For the second set of samples sent February 20, 1985 the sample numbers given are those used by the Cape Breton Coal Research Laboratory (Comp. refers to a composited pair; i.e. comp 7-31 = CBCRL 85-7 + CBCRL 85-31).

SAMPLE NUMBER	SAMPLE DESCRIPTION
1. 84-129-01	Bare millipore filter
2. 84-129-02	Lingan 5W wall
3. 84-129-03	Lingan 9 east - Nov.30/84
4. 84-129-04	Lingan 9 east - Dec. 3/84
5. 84-129-05	Lingan
6. 84-129-06	Lingan 5W wall
7. 84-129-07	Devco stone dust
8. 84-129-08	C-15 Dec.18/84
9. 84-129-09	C-14 Dec.18/84
10. 84-129-10	J-1 Dec.18/84
11. 84-129-11	MSA cellulose membrane
12. 84-129-12	Whatman glass microfibre

NUCLEAR ACTIVATION SERVICES LIMITED

1280 MAIN STREET WEST, HAMILTON, ONTARIO   L8S 4K1

PHONE (416) 527-5666   TELEX 06-986947

CERTIFICATE OF ANALYSIS

TO: ATLANTIC COAL INSTITUTE
ATTN: DAVID O'NEIL
P.O. BOX 1594
SYDNEY, NOVA SCOTIA
B1P 6R9

CUSTOMER NO. 432

DATE SUBMITTED
28-JAN-85

REPORT: 3908

FILE NUMBER: 5574

12 SAMPLES WERE ANALYZED

WERE ANALYZED AS FOLLOWS:

ELEMENTS	UNITS	DETECTION LIMIT	ELEMENTS	UNITS	DETECTION LIMIT
U	PPM	0.0050	TH	PPM	0.0100
NA	PPM	1.0000	CA	%	0.0500
SC	PPM	0.0050	CR	PPM	0.1000
FE	%	0.0010	CO	PPM	0.0100
NI	PPM	5.0000	ZN	PPM	1.0000
AS	PPM	0.0500	SE	PPM	0.1000
BR	PPM	0.0500	MO	PPM	0.2000
SB	PPM	0.0050	CS	PPM	0.0500
BA	PPM	10.0000	LA	PPM	0.0010
HF	PPM	0.0100	TA	PPM	0.0500
W	PPM	0.5000	AU	PPB	5.0000
CE	PPM	0.1000	ND	PPM	3.0000
SM	PPM	0.0050	EU	PPM	0.0500
TB	PPM	0.1000	YB	PPM	0.0050
LU	PPM	0.0050	K	PPM	500.0000
SR	PPM	10.0000	RB	PPM	0.5000

COMMENTS:

CONTINUATION OF WORK ORDER NUMBER 5112

DATE 09-MAY-85

NUCLEAR ACTIVATION SERVICES LIMITE

CERTIFIED BY ....

*** UNLESS INSTRUCTED OTHERWISE WE WILL DISCARD ALL SAMPLES ***
IRRADIATED SAMPLES AFTER 30 DAYS. ANY OTHER MATERIAL AFTER 120 DAYS.

NUCLEAR ACTIVATION SERVICES LIMITED

DATE: 09-MAY-85 REPORT: 3908 FILE NUMBER: 5574 PAGE: 1

SAMPLE NUMBERS

ELEMENT & UNITS	84-129-01	84-129-02	84-129-03	84-129-04	84-129-05	84-129-06
U PPM	<0.005	0.024	<0.017	0.012	0.020	<0.005
TH PPM	<0.01	0.01	0.16	<0.01	0.02	0.02
NA PPM	16	1000	<1	1000	1000	980
CA %	<0.05	<0.05	<0.17	<0.05	<0.05	<0.05
SC PPM	<0.005	0.007	0.011	0.008	0.009	0.008
CR PPM	0.6	0.3	0.6	0.4	0.4	0.3
FE %	0.002	<0.001	<0.001	0.003	<0.001	<0.001
CO PPM	0.05	0.04	0.06	0.03	0.07	0.05
NI PPM	<5	<5	<40	<5	<5	6
ZN PPM	1	480	390	490	490	460
AS PPM	<0.05	0.05	<0.05	<0.05	0.06	<0.05
SE PPM	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
BR PPM	1.0	0.75	0.42	0.39	0.36	<0.05
MO PPM	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2
SB PPM	0.012	0.057	<0.034	0.061	0.055	0.057
CS PPM	<0.05	<0.05	0.34	<0.05	<0.05	<0.05
BA PPM	<10	990	710	960	960	930
LA PPM	0.005	0.063	<0.182	0.073	0.065	0.062
HF PPM	0.01	0.02	<0.09	0.04	0.01	0.02
TA PPM	<0.05	<0.05	0.32	<0.05	<0.05	<0.05
W PPM	<0.5	<0.5	0.6	<0.5	<0.5	<0.5
AU PPB	6	14	<5	<5	<5	<5
CE PPM	<0.1	0.1	<0.1	0.1	0.1	0.1
ND PPM	<3	<3	<3	<3	<3	<3
SM PPM	<0.005	0.006	<0.005	0.008	0.009	0.008
EU PPM	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
TB PPM	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
YB PPM	<0.005	0.009	<0.005	0.007	0.010	0.008
LU PPM	<0.005	<0.005	0.006	<0.005	<0.005	<0.005
K PPM	<500	2000	1700	<500	1700	1400
SR PPM	<10	<10	<10	<10	<10	<10
RB PPM	<0.5	1.5	12	1.6	1.2	1.2

NUCLEAR ACTIVATION SERVICES LIMITED

DATE: 09-MAY-85 REPORT: 3908 FILE NUMBER: 5574 PAGE: 2

SAMPLE NUMBERS

ELEMENT & UNITS	84-129-07**	84-129-08**	84-129-09**	84-129-10**	84-129-11**	84-129-12**
U PPM	0.517	0.005	0.011	0.011	0.026	0.454
TH PPM	0.11	<0.01	0.01	0.02	0.02	1.6
NA PPM	25	38	51	49	79	150
CA %	- - - -	<0.05	<0.05	<0.05	0.17	1.30
SC PPM	0.17	0.011	0.020	0.027	0.055	0.58
CR PPM	1.2	0.7	3.3	0.8	10	1.7
FE %	- - - -	<0.001	0.005	0.007	0.015	0.021
CO PPM	0.21	0.06	0.12	0.09	0.23	0.11
NI PPM	- - - -	<5	<5	<5	<5	<5
ZN PPM	- - - -	12	5	4	10	35
AS PPM	0.28	<0.05	0.10	0.10	0.05	0.2
SE PPM	<0.1	<0.1	<0.1	<0.1	<0.1	<0.2
BR PPM	0.41	3.3	2.2	2.5	5.2	1.7
MO PPM	0.2	<0.2	<0.2	<0.2	0.6	0.6
SB PPM	0.075	0.048	0.086	0.099	0.23	0.41
CS PPM	0.18	0.05	<0.05	0.06	0.20	1.7
BA PPM	40	<10	<10	<10	<10	340
LA PPM	0.482	0.019	0.038	0.056	0.062	1.35
HF PPM	0.03	<0.01	0.01	0.03	0.16	1.0
TA PPM	<0.05	<0.05	<0.05	<0.05	<0.05	0.17
W PPM	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
AU PPB	<5	<5	5	7	9	<5
CE PPM	1.0	<0.1	0.1	0.1	0.2	2.6
ND PPM	<3	<3	<3	<3	<3	<3
SM PPM	0.103	<0.005	0.006	0.009	0.011	0.143
EU PPM	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
TB PPM	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
YB PPM	0.041	<0.005	<0.005	0.006	0.009	0.160
LU PPM	0.006	<0.005	<0.005	<0.005	<0.005	0.033
K PPM	- - - -	<500	<500	<500	<500	<500
SR PPM	50	<10	<10	<10	<10	170
RB PPM	1.1	0.6	<0.5	<0.5	0.8	1.9

EXPLANATION OF CODES

- - - - - NOT REQUESTED

NUCLEAR ACTIVATION SERVICES LIMITED

1280 MAIN STREET WEST, HAMILTON, ONTARIO   L8S 4K1

PHONE (416) 522-5060   TELEX 06-986947

CERTIFICATE OF ANALYSIS

TO: ATLANTIC COAL INSTITUTE
ATTN: DAVID O'NEIL
P.O. BOX 1594
SYDNEY, NOVA SCOTIA
B1P 6R8

CUSTOMER NO. 402

DATE SUBMITTED
28-JAN-85

REPORT: 3908   FILE NUMBER: 0112

12 SAMPLES

WERE ANALYZED AS FOLLOWS:

ELEMENTS	UNITS	DETECTION LIMIT	ELEMENTS	UNITS	DETECTION LIMIT
MG	%	0.0500	AL	%	0.0010
CL	PPM	10.0000	TI	PPM	5.0000
V	PPM	0.1000	MN	PPM	0.1000
GA	PPM	1.0000	IN	PPB	5.0000
DY	PPM	0.0050	I	PPM	0.1000

DATE 09-MAY-85

NUCLEAR ACTIVATION SERVICES LIMITED

CERTIFIED BY ....

*** UNLESS INSTRUCTED OTHERWISE WE WILL DISCARD ALL SAMPLES ***
IRRADIATED SAMPLES AFTER 30 DAYS. ANY OTHER MATERIAL AFTER 120 DAYS.

```
                         NUCLEAR ACTIVATION SERVICES LIMITED
 DATE: 09-MAY-85      REPORT:   3909    FILE NUMBER:   5112          PAGE:   1

                              S A M P L E   N U M B E R S
 ELEMENT  :          **          **          **          **          **          **
 & UNITS  !  4-129-01** 84-129-02** 34-129-03** 34-129-04** 34-129-05** 34-129-06**
----------+-------------------------------------------------------------------------

 MG %          <0.05       <0.05       <0.05       <0.05       <0.05       <0.05
 AL %          0.331       0.330       0.320       0.300       0.390       0.320
 CL PPM           30          60          90          96          70          70
 TI PPM           <5          73          62          93         <52         <54
 V  PPM          0.3         0.6        <0.6         1.3         1.3         2.3

 MN PPM          0.3         1.7         2.0         2.3         2.0         2.4
 GA PPM           <1           4           4           4           7           2
 IN PPB           <5          42          70          41          46          27
 DY PPM       <0.005       0.095       0.093       0.061       0.055      <0.045
 I  PPM          0.1         3.7         3.7         4.3         3.9         3.2
```

NUCLEAR ACTIVATION SERVICES LIMITED

DATE: 09-MAY-85 REPORT: 3908 FILE NUMBER: 5112 PAGE: 2

SAMPLE NUMBERS

ELEMENT & UNITS	84-129-07**	84-129-08**	84-129-09**	84-129-10**	84-129-11**	84-129-12**
MG %	- - - -	<0.05	<0.05	<0.05	<0.05	0.05
AL %	- - - -	0.009	0.009	0.010	0.003	0.680
CL PPM	60	130	80	60	40	10
TI PPM	- - - -	10	8	13	<5	73
V PPM	7.1	2.5	2.3	1.9	1.3	2.5
MN PPM	550	1.2	0.8	1.5	1.1	5.2
GA PPM	2	1	1	1	1	4
IN PPB	240	<5	5	<5	6	16
DY PPM	0.492	<0.005	0.008	0.007	<0.005	0.190
I PPM	3.0	0.3	<0.1	<0.1	0.1	<0.1

EXPLANATION OF CODES

- - - - - NOT REQUESTED

X-RAY ASSAY LABORATORIES LIMITED
1885 LESLIE STREET, DON MILLS, ONTARIO M3B 3J4
PHONE 416-445-5755 TELEX 06-986947

CERTIFICATE OF ANALYSIS

TO: NUCLEAR ACTIVATION SERVICES
ATTN: DR. ERIC HOFFMAN
1280 MAIN STREET WEST
HAMILTON, ONTARIO
L8S 4K1

CUSTOMER NO. 662

DATE SUBMITTED
29-JAN-85

REPORT 23649

REF. FILE 19250-H5

1 PULP PROJ. 5112

WAS ANALYSED AS FOLLOWS:

	METHOD	DETECTION LIMIT
BE PPM	DCP	1.000
WRMAJ %	WR	0.010
WRMIN PPM	WR	10.000
NI PPM	DCP	0.100
CU PPM	DCP	0.050
ZN PPM	DCP	0.050
AG PPM	DCP	0.050
CD PPM	DCP	0.200
PB PPM	DCP	0.200
ASH %	WET	0.100

DATE 19-FEB-85

X-RAY ASSAY LABORATORIES LIMITED

CERTIFIED BY ....

```
X-RAY ASSAY LABORATORIES 19-FEB-85  REPORT 23649 REF. FILE 19250-H5 PAGE   1

   SAMPLE              BE PPM    NI PPM   CU PPM   ZN PPM
   ------------------------------------------------------------
   84-129-07               <1     15.0     3.50     14.0
```

SAMPLE	AG PPM	CD PPM	PB PPM	ASH %
84-129-07	2.50	<0.2	50.0	99.9

NOTE: NOT SUFFICIENT SAMPLE TO DO
CHECK ON LOW SUM.

```
X     X    RRRRR        A       LL
XX   XX    RR  RR      AAA      LL
 XX XX     RR   RR    AA AA     LL
  XXX      RR  RR    AA   AA    LL
  XXX      RRRRR     AAAAAAA    LL
 XX XX     RR  RR    AA   AA    LL
XX   XX    RR   RR   AA   AA    LLLLLLL
X     X    RR    R   AA   AA    LLLLLLL
```

XRF - WHOLE ROCK ANALYSIS

NUCLEAR ACTIVATION SERVICES
Attn: DR. ERIC HOFFMAN
1280 MAIN STREET WEST
HAMILTON, ONTARIO
L8S 4K1

CUSTOMER No. 662

DATE SUBMITTED
29-JAN-85

REPORT 23649 REF. FILE 19250 DATE REPORTED 19-FEB-85

XRF W. R. A. SUMS INCLUDE ALL ELEMENTS DETERMINED.
FOR SUMMATION ELEMENTS ARE CALCULATED AS OXIDES.

X-RAY ASSAY LABORATORIES 19-FEB-85 REPORT 23649 REFERENCE FILE 19250 PAGE 1

SAMPLE	SIO2	AL2O3	CAO	MGO	NA2O	K2O	FE2O3	MNO	TIO2	P2O5	LOI	SUM
04-129-07	3.09	0.72	51.4	0.54	0.01	0.13	0.26	0.04	0.02	0.02	39.6	95.9

SAMPLE	CR	RB	SR	Y	ZR	NB	BA
84-129-07	<10	10	260	<10	<10	10	<10

NUCLEAR ACTIVATION SERVICES LIMITED

1280 MAIN STREET WEST, HAMILTON, ONTARIO   L8S 4K1

PHONE (416) 522-5666   TELEX 06-986947

CERTIFICATE OF ANALYSIS

TO: ATLANTIC COAL INSTITUTE
ATTN: DAVID O'NEIL
P.O. BOX 1594
SYDNEY, NOVA SCOTIA
B1P 6R8

CUSTOMER NO. 402

DATE SUBMITTED
28-JAN-85

REPORT: 3904   FILE NUMBER: 5552

13 PREPARED SAMPLES

WERE ANALYZED AS FOLLOWS:

ELEMENTS	UNITS	DETECTION LIMIT	ELEMENTS	UNITS	DETECTION LIMIT
U	PPM	0.0100	TH	PPM	0.1000
NA	PPM	20.0000	CA	%	0.1000
SC	PPM	0.0500	CR	PPM	0.2000
FE	%	0.0100	CO	PPM	0.1000
NI	PPM	50.0000	ZN	PPM	3.0000
AS	PPM	0.2000	SE	PPM	0.5000
BR	PPM	0.5000	MO	PPM	0.4000
AG	PPM	0.1000	SB	PPM	0.1000
CS	PPM	0.1000	BA	PPM	20.0000
LA	PPM	0.1000	HF	PPM	0.1000
TA	PPM	0.1000	W	PPM	1.0000
AU	PPB	1.0000	CE	PPM	0.5000
ND	PPM	0.5000	SM	PPM	0.0500
EU	PPM	0.0500	TB	PPM	0.0500
YB	PPM	0.0100	LU	PPM	0.0100
K	PPM	500.0000	SR	PPM	50.0000
RB	PPM	1.0000			

COMMENTS:

CONTINUATION OF WORK ORDER NUMBER 5249

DATE 03-MAY-85

NUCLEAR ACTIVATION SERVICES LIMITED

CERTIFIED BY ....

*** UNLESS INSTRUCTED OTHERWISE WE WILL DISCARD ALL SAMPLES ***
IRRADIATED SAMPLES AFTER 30 DAYS. ANY OTHER MATERIAL AFTER 120 DAYS.

NUCLEAR ACTIVATION SERVICES LIMITED

DATE: 09-MAY-85 REPORT: 3904 FILE NUMBER: 5552 PAGE: 1

SAMPLE NUMBERS

ELEMENT & UNITS	CBCRL** 85-96**	CBCRL** 85-33**	CBCRL** 85-98**	CBCRL** 85-35**	CBCRL 85-8**	CBCRL** 85-62**
U PPM	0.11	0.10	0.12	0.10	0.07	0.01
TH PPM	0.2	0.3	0.2	0.2	0.2	0.1
NA PPM	240	270	290	290	310	240
CA %	<0.1	0.2	<0.1	<0.1	0.1	0.2
SC PPM	0.41	0.50	0.44	0.35	0.41	0.20
CR PPM	6.7	12	3.4	9.6	4.2	6.8
FE %	0.12	0.31	0.15	0.12	0.10	0.10
CO PPM	1.1	1.7	1.2	1.7	2.0	1.6
NI PPM	<50	<50	<50	<50	<50	<50
ZN PPM	40	69	56	65	91	49
AS PPM	0.7	1.1	0.9	1.4	1.6	1.0
SE PPM	0.6	<0.5	<0.5	0.9	<0.5	<0.5
BR PPM	9.0	16	17	16	17	19
MO PPM	0.5	1.3	0.6	2.5	0.9	1.2
AG PPM	0.5	0.6	1.5	2.0	0.5	0.8
SB PPM	2.3	1.4	2.5	2.5	1.7	5.2
CS PPM	0.5	0.6	0.3	0.5	0.5	1.0
BA PPM	30	70	40	70	60	30
LA PPM	1.5	1.0	1.0	[illegible]	0.9	0.5
HF PPM	0.2	0.1	0.2	0.2	0.3	0.2
TA PPM	<0.1	<0.1	0.2	0.2	<0.1	<0.1
W PPM	<1	<1	<1	1	<1	<1
AU PPB	13	270	25	21	25	16
CE PPM	2.3	2.4	2.3	2.1	2.2	0.8
ND PPM	1.0	<0.5	<0.5	<0.5	<0.5	<0.5
SM PPM	0.17	0.17	0.15	0.12	0.14	0.06
EU PPM	0.05	<0.05	0.10	0.12	0.13	<0.05
TB PPM	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
YB PPM	0.05	0.07	0.04	0.05	0.06	0.01
LU PPM	0.01	0.01	0.01	<0.01	0.01	<0.01
K PPM	500	900	500	500	500	<500
SR PPM	<50	<50	<50	<50	<50	<50
RB PPM	3	<2	1	<3	<2	<1

NUCLEAR ACTIVATION SERVICES LIMITED

DATE: 09-MAY-85 REPORT: 3904 FILE NUMBER: 5552 PAGE: 2

S A M P L E N U M B E R S

ELEMENT & UNITS	CBCPL** 35-39**	CBCFL** 85-40**	** CUMP 4-5**	** COMP 76-9**	** COMP 52-53**	** CUMP 73-7.**
U PPM	0.32	0.13	0.05	0.09	0.10	0.18
TH PPM	0.2	0.3	0.2	0.3	0.3	0.5
NA PPM	220	290	200	180	200	230
CA %	<0.1	<0.1	<0.1	0.1	<0.1	<0.1
SC PPM	0.25	0.42	0.30	0.41	0.38	0.75
CR PPM	7.7	10	7.5	7.3	5.9	9.5
FE %	0.06	0.13	0.09	0.09	0.11	0.20
CO PPM	1.4	2.1	1.0	0.8	0.8	1.5
NI PPM	<50	<50	<50	<50	<50	<50
ZN PPM	55	69	78	56	39	36
AS PPM	0.9	1.3	1.0	0.4	0.7	1.5
SE PPM	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
BR PPM	19	24	10	3.4	9.6	9.0
MO PPM	0.9	2.2	<0.4	3.6	0.4	0.8
AG PPM	1.5	<0.1	0.7	0.4	0.7	0.3
SB PPM	0.9	2.6	2.2	1.3	2.7	1.5
CS PPM	0.5	3.3	0.3	0.7	0.3	0.5
BA PPM	60	70	40	30	20	40
LA PPM	0.5	0.9	0.7	1.0	0.9	1.8
HF PPM	0.2	0.2	0.1	<0.1	0.1	0.2
TA PPM	0.2	<0.1	0.1	<0.1	<0.1	<0.1
W PPM	<1	<1	<1	<1	<1	<1
AU PPB	<1	28	13	12	16	15
CE PPM	2.0	2.8	1.8	1.9	2.2	4.3
ND PPM	<0.5	<0.5	<0.5	0.6	<0.5	1.6
SM PPM	0.10	0.15	0.12	0.15	0.14	0.29
EU PPM	0.03	<0.07	0.05	<0.05	0.05	0.08
TB PPM	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
YB PPM	0.02	0.04	0.04	0.06	0.05	0.10
LU PPM	<0.01	0.01	<0.01	0.01	0.01	0.02
K PPM	500	<500	<500	<1300	600	700
SR PPM	<50	<50	<50	<50	<50	<50
RB PPM	1	5	<2	[illegible]	<1	5

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                         NUCLEAR ACTIVATION SERVICES LIMITED

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                              S A M P L E   N U M B E R S

ELEMENT   !           **
& UNITS   ! COMP 7-31**
----------+------------
U  PPM          0.07
TH PPM           0.1
NA PPM           190
CA %            <0.1
SC PPM          0.27

CR PPM           6.5
FE %            0.09
CO PPM           0.7
NI PPM           <50
ZN PPM           110

AS PPM           0.3
SE PPM          <0.5
BR PPM            11
MO PPM          <0.4
AG PPM           0.6

SB PPM           2.1
CS PPM           0.2
BA PPM            30
LA PPM           0.6
HF PPM           0.1

TA PPM          <0.1
W  PPM            <1
AU PPB            15
CE PPM           1.4
ND PPM          <0.5

SM PPM          0.11
EU PPM          0.03
TB PPM         <0.05
YB PPM          0.03
LU PPM          0.01

K  PPM          <500
SR PPM           <50
RB PPM            <1
```

NUCLEAR ACTIVATION SERVICES LIMITED
1280 MAIN STREET WEST, HAMILTON, ONTARIO   L8S 4K1
PHONE (416) 522-5660   TELEX 06-986947

CERTIFICATE OF ANALYSIS

TO: ATLANTIC COAL INSTITUTE
ATTN: DAVID O'NEIL
P.O. BOX 1594
SYDNEY, NOVA SCOTIA
B1P 6R9

CUSTOMER NO. 402

DATE SUBMITTED
23-JAN-85

REPORT: 3904   FILE NUMBER: 5249

15 PREPARED SAMPLES

WERE ANALYZED AS FOLLOWS:

ELEMENTS	UNITS	DETECTION LIMIT	ELEMENTS	UNITS	DETECTION LIMIT
MG	%	0.0500	AL	%	0.0010
CL	PPM	10.0000	TI	PPM	5.0000
V	PPM	0.1000	MN	PPM	0.1000
GA	PPM	1.0000	IN	PPB	5.0000
DY	PPM	0.0050	I	PPM	0.1000

DATE 10-MAY-85

NUCLEAR ACTIVATION SERVICES LIMITED

CERTIFIED BY ....

*** UNLESS INSTRUCTED OTHERWISE WE WILL DISCARD ALL SAMPLES ***
IRRADIATED SAMPLES AFTER 30 DAYS. ANY OTHER MATERIAL AFTER 120 DAYS.

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                         NUCLEAR ACTIVATION SERVICES LIMITED

DATE: 03-MAY-85      REPORT:   3904    FILE NUMBER:   5249            PAGE:   1

                              S A M P L E   N U M B E R S

ELEMENT  !      CBCRL**      CBCRL**      CBCRL**      CBCRL**                **       CBCRL**
E UNITS  !      85-96**      85-33**      85-93**      85-35**CBCRL 85-8**           85-62**
----------+-----------------------------------------------------------------------------------

MG %           <0.05        <0.05        <0.05        <0.05        <0.05        <0.05
AL %           0.170        0.190        0.200        0.160        0.180        0.080
CL PPM           420          480          440          520          480          560
TI PPM           140          130          150          190          120           78
V  PPM            12           22           20           20           21           17

MN PPM            15           24           16           37           19           15
GA PPM             5           11           12           10           12           12
IN PPB            25           20           30           31          110           30
DY PPM         0.098        0.112        0.103        0.101        0.101        0.035
I  PPM           4.1          4.2          5.3          6.0          3.9          5.2
```

NUCLEAR ACTIVATION SERVICES LIMITED

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SAMPLE NUMBERS

ELEMENT & UNITS	CBCPL** 85-39**	CBCPL** 85-40**	** COMP 4-5**	** COMP 76-9**	** COMP 52-53**	** COMP 73-74**
MG %	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
AL %	0.100	0.180	0.120	0.190	0.160	0.320
CL PPM	350	590	400	380	440	450
TI PPM	110	140	93	130	97	210
V PPM	21	23	11	12	12	13
MN PPM	14	22	14	15	12	30
GA PPM	10	14	7	6	7	8
IN PPB	60	60	<5	32	5	<5
DY PPM	0.063	0.073	0.075	0.098	0.091	0.206
I PPM	3.4	3.7	1.7	2.9	2.7	3.1

```
                 NUCLEAR ACTIVATION SERVICES LIMITED
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                         S A M P L E   N U M B E R S

ELEMENT  !          **
& UNITS  ! COMP 7-31**
---------+------------
MG %          <0.05
AL %          0.120
CL PPM          420
TI PPM           92
V  PPM           10

MN PPM           12
GA PPM            5
IN PPB           18
DY PPM        0.070
I  PPM          2.3
```